Jan Delaval please

Access DB# 72598

SEARCH REQUEST FORM

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****

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for Production of Sterols

Inventors (please provide full names): Mehdi BONAKDAR et al.

Earliest Priority Filing Date: 8/7/2001

**For Sequence Searches Only* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.*

(a) oil ~~from~~ Tall oil, soybean oil etc (See cl. 3)

(b) transesterification

(c) Removal of alcohol, catalyst etc

(d) transesterification of sterol esters = to get free sterols.

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Sequence Systems

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Other (specify)

PTO-1590 (8-01)

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MOST RECENT DERWENT UPDATE           200251   <200251/DW>
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=> d all abeq tech abex tot l58

L58  ANSWER 1 OF 34  WPIX (C) 2002 THOMSON DERWENT
AN   2002-241728 [29]   WPIX
DNC  C2002-072731
TI   Enrichment and isolation of sterols and/or tocopherols from
     mixtures of fats and/or fat derivatives also containing glycerides
     involves repeated hydrolysis, separation and distillation
     stages.
DC   D23 E13 E15
IN   ALBIEZ, W; KOZAK, W G; LOUWEN, T
PA   (COGN-N) COGNIS DEUT GMBH
CYC  21
PI   WO 2002012222 A1 20020214 (200229)* DE   14p    C07D311-72
        RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR
         W: BR US
     DE 10038457   A1 20020221 (200229)              C07J009-00      <--
ADT  WO 2002012222 A1 WO 2001-EP8877 20010801; DE 10038457 A1 DE 2000-10038457
     20000807
PRAI DE 2000-10038457 20000807
IC   ICM  C07D311-72; C07J009-00
AB   WO 200212222 A UPAB: 20020508
     NOVELTY - Enrichment and isolation of sterols (I) and/or
     tocopherols (II) from mixtures of fats and/or fat derivatives containing
     (I)/(II) and glycerides (III), comprises:
          (a) hydrolyzing (III) to free fatty acids (IVA)
     and glycerol;
          (b) separating water containing glycerol;
          (c) distilling off (IVA) and readily volatile,
     unsaponifiable components;
          (d) hydrolyzing the distillation residue once or
     more to convert (I) esters to free fatty acids
```

(IVB) and (I);
(e) separating water; and
(f) **distilling** off (IVB).
USE - None given.
ADVANTAGE - The most important natural sources of tocopherols (II) are not **vegetable oils** but steam **distillates** obtained in deodorization of **vegetable** and animal **oils**. . Normally, the free **acids** are **esterified** with a **lower alcohol** in the presence of a basic catalyst but the catalyst often contains metal and can pollute the environment. The present process allows simultaneous recovery of **sterols** (I) and (II) from many different starting materials, without using harmful solvents or catalysts. It gives good yields, even from starting materials of low concentration, and can be carried out economically on the technical scale.
Dwg.0/0

FS CPI
FA AB; DCN
MC CPI: D10-B01; **E01**; E06-A01; **E11-Q01**
TECH UPTX: 20020508
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Composition: The starting material is a mixture of free **fatty acids**, (I) **esters** and **glycerides**, preferably a **residue** from **distillation** of **fatty acids**, especially a steam **distillate**, more especially a steam **distillate** of **rapeseed oil**, **sunflower oil**, **palm oil**, **palm kernel oil**, **coconut oil**, **soya oil**, maize **oil** and/or cottonseed **oil**.

ABEX
EXAMPLE - A soya oil steam distillate contained 31.5 wt.% fatty acids, 12.2 wt.% tocopherols (II), 8.2 wt.% sterols (I), 19.5 wt.% (I) esters, 17.0 wt.% glycerides and 11.2 wt.% other compounds. 1000 g steam distillate and 500 g deionized water were heated at 220 degreesC for 3 hours in a stirred autoclave. After cooling, water containing glycerol was removed in a phase separator. This gave 970 g dried product containing 49.6 wt.% fatty acids, 12.6 wt.% (II), 12.0 wt.% (I), 14.3 wt.% (I) esters and 11.5 wt.% other. The fatty acids were distilled off in a distillation column with a thin film evaporator, operating at a top pressure of 1.8 mbar and heating medium at 270 degreesC. 1000 g charge gave a distillate free from (II) and (I) and 450 g sump product containing 4.4 wt.% fatty acids, 26.9 wt.% (II), 25.7 wt.% (I), 30.5 wt.% (I) esters and 12.4 wt.% other. 400 g sump product and 400 g deionized water were heated at 220 degreesC for 6 hours in a stirred autoclave. After cooling, water was removed in a phase separator. The dried product contained 14.9 wt.% fatty acids, 26.9 wt.% (II), 42.2 wt.% (I), 3.6 wt.% (I) esters and 12.4 wt.% other. Distillation as above gave fatty acids as distillate and a residue containing 1.5 wt.% fatty acids, 31.2 wt.% (II), 48.8 wt.% (I), 4.2 wt.% (I) esters and 14.3 wt.% other. Over 90% of free (I) and/or 95% of (II) from the mixtures of fats and/or fat derivatives could be concentrated in this way by repeated hydrolysis.

L58 ANSWER 2 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN **2002-139889** [18] WPIX
DNC **C2002-043110**
TI Extracting purified tocopherols and **sterols**, for chemical or pharmaceutical use, from mixtures containing **fatty acids**, by **esterifying** the **acids** with trimethylol propane and two-stage molecular **distillation**.
DC B02 E13
IN ELOY MURO, A
PA (VITA-N) VITAE-CAPS SA
CYC 21

PI WO 2002000640 A1 20020103 (200218)* ES 28p C07D311-72
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: CA JP US
ADT WO 2002000640 A1 WO 2000-ES237 20000705
PRAI ES 2000-1604 20000628
IC ICM C07D311-72
ICS C11B007-00
AB WO 200200640 A UPAB: 20020319
NOVELTY - A method for extraction and purification of natural tocopherols (I) and **sterols** (II), utilizing **esterification** with trimethylol propane (TMP), involves: (i) **esterifying** **fatty acids** with TMP in presence of hypophosphorous **acid** as catalyst and **methanol** as diluent) in a ratio of 1 to 7; (ii) centrifuging or filtering at -30 deg. C; (iii) separating the **fatty acid**-TMP **esters** from (I), (II) and hydrocarbons by molecular **distillation**; and (iv) carrying out further molecular **distillation** under specific conditions to purify (I).
USE - The process is useful for recovering pure (I) (i.e. vitamin E and its homologs) and (II) (especially the **phytosterols** **ergosterol, stigmasterol, campesterol** and **sitosterol**) from natural **vegetable oil** sources, especially from deodorization **distillates** obtained in the as by-products in the purification of **vegetable oils** (e.g. **sunflower, soya, rape** or maize **oil**). (I) and (II) are useful in the chemical and pharmaceutical industries. In particular (I) are antioxidants for use in food, cosmetic or pharmaceutical products or in paints based on natural **oils**; and (II) are starting materials for drugs, especially steroid hormones such as corticosteroids or gestagens (e.g. **stigmasterol** can be converted easily into **progesterone**).
ADVANTAGE - The **fatty acids** present in the starting material are selectively converted into high molecular weight **esters** of low volatility, to facilitate separation of (I) and (II) by selective **distillation**. Typically (I) can be recovered in more than 80% yield from **residues** containing (I) at less than 6%. The molecular **distillation residues**, containing ca. 50% TMP **esters, fatty acid** di- and **triglycerides** and a small amount of **monoglycerides**, may be utilized as viscosity regulators in lubricating **oils**.
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B04-J02; B06-A01; B11-B; **E01**; E06-A01; **E11-Q01**

L58 ANSWER 3 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2001-432315 [46] WPIX
CR 2000-564724 [52]
DNC C2001-130726
TI Separation of a **sterol** or **sterol ester**, e.g. the food additive beat-**sitosterol**, from crude **tall** **oil** comprises fractionating the **oil** such that the residue fraction, including the **sterol**, does not exceed a specified temperature.
DC B01 **D23** E15
IN HUIBERS, D T A; ROBBINS, A M; SULLIVAN, D H
PA (ARIZ) ARIZONA CHEM CORP; (ARIZ) ARIZONA CHEM CO
CYC 1
PI US 2001007906 A1 20010712 (200146)* 11p C07J075-00
US 6414111 B2 20020702 (200248) C09F001-00
ADT US 2001007906 A1 CIP of US 1998-143959 19980831, Div ex US 1998-153728 19980915, Div ex US 2000-618121 20000717, US 2001-782869 20010214; US 6414111 B2 CIP of US 1998-143959 19980831, Div ex US 1998-153728 19980915,

Div ex US 2000-618121 20000717, US 2001-782869 20010214
FDT US 2001007906 A1 Div ex US 6107456; US 6414111 B2 Div ex US 6107456
PRAI US 1998-153728 19980915; US 1998-143959 19980831; US 2000-618121 20000717; US 2001-782869 20010214
IC ICM C07J075-00; C09F001-00
ICS B01D001-00; B01D001-22
AB US2001007906 A UPAB: 20020730
NOVELTY - Separation of a **sterol** or **sterol ester** from crude **tall oil** comprises fractionating the **oil** into a residue fraction and a volatile fraction. During fractionation, the temperature of the residue fraction, which includes the **sterol** (**ester**), does not exceed 290 deg. C.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for:

(1) A residue fraction comprising at least about 15% rosin **acids**.

(2) A method for separating unsaponifiable material from a **tall oil** stream comprising saponifying the stream with a mixture of sodium and potassium hydroxides to form sodium and potassium salts of **fatty acids**, rosin **acids**, or both, evaporating the unsaponifiable material, and acidulating the unevaporated salts

USE - Separation of **sterol** (**ester**) from crude **tall oil**. beta -**Sitosterol** promotes the reduction of circulating **cholesterol** in humans, and is a food additive.

ADVANTAGE - More than 50% of the **sterols** is recovered.

DESCRIPTION OF DRAWING(S) - The figure is a schematic drawing of an apparatus for separating **sterols** from crude **tall oil**.

Crude **tall oil** 102
Contactor regions 103-106
Flasher 107
Reboiler 118
Dwg.1/3
FS CPI
FA AB; GI; DCN
MC CPI: B01-D02; B04-C03D; B04-J02; D03-H01T2; **D10-A04**; **E01**; **E11-Q01**
TECH UPTX: 20010815
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Process: The temperature of the crude **tall oil** fractionation feed is 300-310 degreesC. Fractionation is carried out in a thin film evaporator at below 250 degreesC. The temperature of the residue fraction is 250-270 degreesC. The time of separating the residue fraction from the volatile fraction does not exceed one hour, after which the residue fraction is allowed to cool. The residue fraction comprises at least 15% rosin acids. The method further comprises **esterification** of the **sterols** in the crude **tall oil** before or during fractionation. The degree of **esterification** is preferably greater than 50%. The **sterols** are separated from the non-**sterol** components of the residue fraction. The residue fraction is saponified to recover free **sterols**. Saponification is carried out with a mixture of sodium and potassium hydroxides. The saponified residue fraction is evaporated and extracted with a solvent to provide an extract containing **sterols**. The **tall oil** stream preferably is a **tall oil** pitch stream.
ABEX
EXAMPLE - 48,000 Pounds per hour of crude tall oil (CTO) (102) were fed through feed flasher (107) set at 316 degreesC. The CTO exited at 308 degreesC and entered column (101). The CTO was fractionated in contactor regions (103)-(106), of which contactor (105) had a temperature of 282-276 degreesC and contactor (104) had a temperature of 251-276 degreesC. The

residue fraction that left the column via tube (123) at 316 degreesC and coursed through the reboiler (118) attained a temperature of 330 degreesC as it exited the reboiler and re-entered the column. A residue fraction was removed from the column at outlet (122), at 311 degreesC. 5,225 Pounds per hour of residue fraction left column (101), resulting in a residue yield of 10.9%. The total beta-sitosterol content of the CTO feed was 2.01% (965 lbs/hour). (beta-Sitosterol is the principal sterol in CTO, constituting about 80% of the five sterols and stanols identified). The content of beta-sitosterols in the residue fraction was 5.66% (296 lbs/hour) of which 0.48% was free and 5.18% was esterified The residue fraction included 3.8% stigmastadiene (196 lbs/hour), which is a dehydration product of beta-sitosterol. Thus, 31% of the beta-sitosterol in the CTO feed was recovered in the residue and 22% was dehydrated in the residue to stigmastadiene.

L58 ANSWER 4 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2001-335820 [35] WPIX
DNC C2001-103743
TI Production of **phytosterols**, in high yield and purity, from crude **fatty acid** products, without the need for repeated recrystallization steps to obtain the pure product.
DC B01 D13 D21 **D23**
IN HATTORI, Y; HORIO, M; KONO, J
PA (KAOS) KAO CORP
CYC 21
PI WO 2001032682 A1 20010510 (200135)* EN 23p C07J009-00 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR
W: US
JP 2001131197 A 20010515 (200143) 4p C07J009-00 <--
JP 2001131199 A 20010515 (200143) 5p C07J075-00
ADT WO 2001032682 A1 WO 2000-JP7753 20001102; JP 2001131197 A JP 1999-313619 19991104; JP 2001131199 A JP 1999-313620 19991104
PRAI JP 1999-313620 19991104; JP 1999-313619 19991104
IC ICM **C07J009-00**; C07J075-00
ICS C11B013-00
AB WO 200132682 A UPAB: 20010625
NOVELTY - **Phytosterols** are product from crude **fatty acid** products by
(i) crystallization of the **phytosterols** using a water/organic solvent mixture; or
(ii) dissolution of the **phytosterols** from the **fatty acid** product into a **lower alcohol**.
DETAILED DESCRIPTION - Production of a **phytosterol**, comprises:
(a) bringing a crude **fatty acid** (which is derived from a **vegetable** fat and/or **oil** and includes a **phytosterol**) into contact with a mixture of an organic solvent and water, to crystallize the **phytosterol**, and separating of the crystals from the solvent mixture; or
(b) mixing a crude **fatty acid ester** (which is derived from a **vegetable** fat and/or **oil** and includes a **phytosterol** and a **fatty acid ester**) with a **lower alcohol**; allowing the mixture to stand at 1-40 deg. C to precipitate crystals which include the **fatty acid ester**, and separating the crystals to give a **lower alcohol** solution which includes the **phytosterol**.
ACTIVITY - Antilipemic.
MECHANISM OF ACTION - None given.
USE - The above processes are useful for production of **phytosterols**. **Phytosterols** are capable of lowering **cholesterol** levels in blood, and can also be used as starting materials for production of **cholesterol** substitutes or as

emulsifiers or emulsion stabilizers in cosmetics and foods.
ADVANTAGE - The processes are easy to carry out and give highly pure **phytosterols** in high yields. They remove the need for repeated recrystallization of products to give highly pure **phytosterols**
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B01-D02; B12-M09; B14-D02A2; B14-F06; D03-H01N; D03-H01T2; D08-B13; **D10-A04**
TECH UPTX: 20010625
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred process: The **vegetable** fat is a **palm kernel oil**, a **coconut oil** or a **palm oil**. In process (a), the amount of water in the solvent mixture is at least 1 wt.% of the mixture. The organic solvent has a relative dielectric constant of not less than 17 at 25degreesC. This process also comprises allowing a mixture of the crude **fatty acid** product and a **lower alcohol** to stand at 1-40degreesC to precipitate crystals which include a **fatty acid ester**. In process (b), the amount of **lower alcohol** is 2 wt.%, as compared to the weight of the crude **fatty acid ester**. Processes (a) and (b) can be carried out in combination with each other.
ABEX
EXAMPLE - A crude fatty acid methyl ester product (5,000 g) derived from palm kernel oil was distilled to give a distillate (4,900 g) and a residue (100 g). The residue was treated with methanol (100 g) and potassium hydroxide (1.0 g). The mixture was refluxed for 2 hours so that a transesterification reaction occurred. The resulting crude fatty acid methyl ester product obtained contained 6-28C fatty acid methyl esters (68%) and phytosterols (5.7%), the percentages being based on the oil material produced and excluding methanol present. Water (55%; based on the amount of residual methanol) was added to the solution after the reaction, keeping the temperature of the solution at 5degreesC, and crystallization was carried out. The precipitated crystals were recovered at 5degreesC using a vacuum filter. Methanol (100 g) was added to the crystals, and the crystals were dissolved at 60degreesC. The solution was then cooled to 5degreesC, and the resulting crystals were removed by filtration. The crystals were dried in air at 110degreesC for 12 hours. The recoverable ratio of phytosterols by this method was 69%, and the purity of the phytosterol product was 68%.

L58 ANSWER 5 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2001-136445 [14] WPIX
DNC C2001-039915
TI Production of free **sterols** from material comprising **sterols** and saponifiable compounds uses horizontal rotary thin-film apparatus having two external jackets to provide reaction and evaporation zones.
DC B01 **D23** E15
IN ALASTI, P
PA (ARTZ) ARTISAN IND INC
CYC 1
PI US 6160143 A 20001212 (200114)* 6p C11B003-00
ADT US 6160143 A US 2000-507405 20000218
PRAI US 2000-507405 20000218
IC ICM C11B003-00
AB US 6160143 A UPAB: 20010312
NOVELTY - Production of free **sterols** from a material comprising **sterols** and saponifiable compounds uses a horizontal rotary thin-film apparatus (I) having two external jackets to provide an internal thin-film reaction zone for saponifying the saponifiable compounds and an internal thin-film evaporation zone for concentrating the saponification

mixture.

DETAILED DESCRIPTION - Process for the concentration and separation of free **sterols** from a feed source comprising **sterol** -containing material and saponifiable material comprises:

(a) introducing the feed source into the reaction zone of (I);

(b) introducing an aqueous alkali solution into the reaction zone to saponify the saponifiable material and form a mixture of soap, free **sterols**, water and light hydrocarbons;

(c) heating the mixture in the evaporation zone to remove water and light hydrocarbons;

(d) withdrawing a slurry of soap and free **sterols** from the evaporation zone; and

(e) heating the slurry to separate the **sterols** and soap.

USE - The process is especially useful for recovering **sterols** , preferably **sitosterol**, **campesterol** and **stigmasterol**, from a natural oil or fat deodorizer stream.

ADVANTAGE - **Sterol** recovery yields of more than 90 % can be achieved.

DESCRIPTION OF DRAWING(S) - The figure is a schematic illustration of the process, in which the thin-film apparatus, e.g. a Rototherm (RTM) evaporator, has a first heating jacket surrounding the reaction zone and a second heating jacket surrounding the evaporation zone.

Thin-film apparatus 12

First jacket 16

Second jacket. 18

Dwg.1/1

FS CPI

FA AB; GI; DCN

MC CPI: B01-D01; D10-A05A; **E01**; **E11-Q01**

TECH UPTX: 20010312

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Process: The alkali is sodium hydroxide or potassium hydroxide. The reaction zone is operated at 170-210 degrees C with a residence time of 30-120 s. The evaporation zone is operated at 250-290 degrees C. The slurry is heated at 280-320 degrees C and 1-10 mm Hg.

L58 ANSWER 6 OF 34 WPIX (C) 2002 THOMSON DERWENT

AN 2001-031682 [04] WPIX

DNC C2001-009597

TI Isolation of **sterols** from neutral substances using hydrocarbon and alcohol based solvent systems.

DC B01 D13 **D23** E15 F09

IN HAMUNEN, A; KARINEN, P

PA (STER-N) STEROL TECHNOLOGIES LTD

CYC 92

PI WO 2000064923 A1 20001102 (200104)* EN 27p C07J009-00 <--

RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL OA PT SD SE SL SZ TZ UG ZW

W: AE AG AL AM AT AU AZ BA BB BG BR BY CA CH CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW

AU 2000039841 A 20001110 (200109) C07J009-00 <--

ADT WO 2000064923 A1 WO 2000-IB541 20000427; AU 2000039841 A AU 2000-39841 20000427

FDT AU 2000039841 A Based on WO 200064923

PRAI US 1999-131306P 19990427

IC ICM **C07J009-00**

ICS C11B013-00

AB WO 200064923 A UPAB: 20010118

NOVELTY - Isolation of **sterols** from neutral substances using hydrocarbon and alcohol based solvent systems.

DETAILED DESCRIPTION - Method for separating **sterols** from

neutral substances containing the **sterols** comprises:

(a) providing a hydrocarbon fraction containing the neutral substances;

(b) subjecting the hydrocarbon fraction to a first purification step to obtain a crude **sterol** product;

(c) subjecting the crude **sterol** product to a second purification step to obtain a solid product enriched in **sterols**;

(d) optionally subjecting the solid product enriched in **sterols** to a further purification step comprising at least one of crystallization, washing and/or recrystallization; and optionally

(e) one or more intermediate washing steps between any of steps (a)-(d) where at least one of the steps (b)-(d) is carried out using a first solvent comprising a hydrocarbon and at least another of the steps (b)-(d) is carried out using a second solvent system comprising a 1-6C alkanol and the second solvent system is more polar than the first solvent.

ACTIVITY - None given.

MECHANISM OF ACTION - None given.

USE - **Sterols** are used in pharmaceuticals and foods.

ADVANTAGE - The method gives high yields of high quality and high purity **sterols**.

Dwg.0/0

FS CPI

FA AB; DCN

MC CPI: B01-D02; D03-H01; **D10-A04**; **E01**; **E11-Q01** ; F05-A02B; F05-A02C

TECH UPTX: 20010118

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: The first solvent comprises a 1-6C alkanol and water, preferably in a weight ratio of at least 2.3:1. The hydrocarbon, alkanol and water are present in a weight ratio of 1.5-5: less than 0.5: ; less than 1. In the step which uses the first solvent system, the neutral substances, **sterol** and first solvent are present in a weight ratio of 1:1.5-6.5. The second solvent comprises a hydrocarbon and water. The alkanol and the hydrocarbon are present in a weight ratio of at least 1.5:1 and the alkanol, hydrocarbon and water are present in a weight ratio of 4-20:1-2:less than 1. In the step using the second solvent the neutral substances, **sterol** and second solvent are present in a weight ratio of 1:5-23. The hydrocarbon fraction is provided by extracting a soap with a hydrocarbon to provide a hydrocarbon phase and a soap phase and separating the phases, or the soap is extracted with an organic hydrocarbon, the phases are separated, the organic phase is evaporated to dryness and the neutral substances are dissolved in a hydrocarbon.

The hydrocarbon in the first solvent comprises hexane, heptane, octane, cyclohexane and/or methylcyclohexane. The alkanol is preferably **methanol**. The first purification step comprises optionally crystallizing the crude **sterol** product from the hydrocarbon fraction using the first or second solvent, optionally followed by washing the crude **sterol** with the first or second solvent and the second purification step comprises optionally crystallizing the solid product enriched in **sterols** from the first or second solvent system, optionally followed by washing the solid product enriched in **sterols** with the first or second solvent system.

ABEX

EXAMPLE - The unsaponifiables from Pinus taeda in a hydrocarbon solvent comprising a mixture of aliphatic and cycloaliphatic hydrocarbons, LIAV110, delivered by Neste Oy. The mixture was brought directly from the soap extraction which was performed at 170 degrees C and 18 bar pressure. Solids content of the hydrocarbon phase was 11.3 % and the sterols content was 35 % of the solids. The hydrocarbon phase was allowed to cool slowly to 20 degrees C. The precipitated sterols were filtered and washed with fresh solvent. When 100 g dry neutrals were used as starting material, 22.5 g sterol blend comprising sitosterol, sitostanol, campesterol and

campestanol and nonelutable impurities were obtained. 2 l of the above hydrocarbon phase was put into an autoclave and 1 l water was added. The autoclave was closed and the temperature was raised to 130 degrees C with stirring. After 5 minutes at 130 degrees C the stirring was stopped and the lower water phase was separated through a water cooled sampling bomb. The hydrocarbon phase was allowed to cool to 20 degrees C and the crystalline material was filtered and washed with fresh hydrocarbon. The purity of the sterol was 82 % and the yield was 23 g/100 g feed natural substances.

L58 ANSWER 7 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2001-007094 [01] WPIX
DNC C2001-001751
TI Separation of neutral substances, especially **sterols**, from soap comprises heating with water and hydrocarbon solvent and separating.
DC B01 D13 D21 **D23** E15 F09
IN HAMUNEN, A
PA (STER-N) STEROL TECHNOLOGIES LTD
CYC 93
PI WO 2000065004 A1 20001102 (200101)* EN 27p C11B013-00
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL OA PT SD SE SL SZ TZ UG ZW
W: AE AG AL AM AT AU AZ BA BB BG BR BY CA CH CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW
AU 2000038328 A 20001110 (200109) C11B013-00
BR 2000010045 A 20020312 (200226) C11B013-00
EP 1190025 A1 20020327 (200229) EN C11B013-00
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT RO SE SI
ADT WO 2000065004 A1 WO 2000-IB542 20000427; AU 2000038328 A AU 2000-38328 20000427; BR 2000010045 A BR 2000-10045 20000427, WO 2000-IB542 20000427; EP 1190025 A1 EP 2000-917241 20000427, WO 2000-IB542 20000427
FDT AU 2000038328 A Based on WO 200065004; BR 2000010045 A Based on WO 200065004; EP 1190025 A1 Based on WO 200065004
PRAI US 1999-131304P 19990427
IC ICM C11B013-00
AB WO 200065004 A UPAB: 20001230
NOVELTY - Separating neutral substances (NSs), particularly **sterols**, from a soap comprises heating with water and a hydrocarbon solvent, separating the hydrocarbon phase, and separating the NSs from the hydrocarbon phase.

DETAILED DESCRIPTION - Separating neutral substances (NSs) from a soap comprises:

(a) heating a mixture comprising: the soap containing the NSs; water, optionally containing sodium sulfate; and a 1-10C hydrocarbon solvent; to at least 140 deg. C, to obtain a soap phase and a hydrocarbon phase containing the NSs, in a closed system under pressure at least equal to the vapor pressure of the mixture at the temperature used in the heating step;

(b) separating the hydrocarbon phase from the soap phase; and

(c) optionally separating the NSs from the hydrocarbon phase.

USE - The method is useful for separating NSs from soaps such as pulping soap obtained as a by-product in the production of sulfate cellulose or soaps obtained from deodorized **distillates** of **plant oils**. The method can be used for separating NSs such as **sterols**, terpene alcohols or fatty alcohols (claimed). The neutral substances can be used in e.g. the food, pharmaceutical and cosmetic industry.

ADVANTAGE - The method allows for regeneration of the solvent, and the problems associated with the use of long chain hydrocarbons are avoided.

Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B01-D01; B01-D02; B04-J02; B10-E04D; B10-J02; D03-H01; D08-B; **D10-A04**; D10-B01; **E01**; E10-E04E5; E10-J02C3; F05-A02C
TECH UPTX: 20001230
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Process: The hydrocarbon solvent is hexane, heptane, octane, cyclohexane and/or methylcyclohexane. The temperature of the process is 140-190 (especially 165-190) degreesC. Preferably the soap, the water and the hydrocarbon solvent are provided in the mixture in a weight ratio of :1more than1:more than1 (especially 1:2-3-4-5), based on the dry weight of the soap. The method may further comprise reducing the separated hydrocarbon phase by evaporation or washing the separated or reduced hydrocarbon phase with water. Washing is at at least 80 (especially at least 130) degreesC under pressure. The separating step comprises evaporating the hydrocarbon phase to dryness. The neutral substance is a terpene alcohol, fatty alcohol or especially a **sterol**. The hydrocarbon phase may additionally contain other neutral substances from the soap apart from **sterols**, and step (c) further comprises purifying the **sterols** from the other neutral substances by dissolving the other neutral substances in a solvent mixture comprising methyl ethyl ketone, a 1-6C alkanol and water, and crystallizing the **sterols** from the solvent mixture .The method further comprises washing the **sterol** crystals with a solvent. Alternatively step (c) comprises crystallizing the **sterols** from the hydrocarbon phase by cooling the hydrocarbon phase and separating the formed crystals from the hydrocarbon phase, or reducing the hydrocarbon phase by evaporation, mixing the reduced hydrocarbon phase with a 1-6C alkanol and optionally water, and crystallizing the **sterols** from the mixture. The 1-6C alkanol is preferably **methanol**.
ABEX
EXAMPLE - Soap originated from Pinus radiata pine wood pulp was extracted in a closed reactor as batch extraction. The soap contained 15% unsaponifiables calculated from the soap solids and the unsaponifiables contained 35% sterols. 500g Soap (solids content 60%) was mixed with 400g water and 900g hydrocarbon solvent LIAV110 (a mixture of straight chain, branched chain and cycloaliphatic saturated hydrocarbons, mainly 6-8C hydrocarbons) and the temperature was elevated to 150 degreesC. After 5 minutes mixing (300 rpm), the phases were allowed to separate for a few minutes. The lower phase was drained out slowly from the bottom valve through a Liebig type water cooled bomb. No clear intermediate (rag) layer was detected between the lower soap layer and upper hydrocarbon layer. When evaporated to dryness, 36g (yield 80%) unsaponifiables was recovered from the hydrocarbon layer. It contained 12.3g sterols, which was 78% of the sterols in the feed soap.

L58 ANSWER 8 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN **2000-564724** [52] WPIX
CR 2001-432315 [46]
DNC **C2000-168197**
TI **Tall oil** processing for separation of component **sterols** involves fractionation of the crude **tall oil** into volatile and residue fractions.
DC D13 E15 H02
IN HUIBERS, D T A; ROBBINS, A M; SULLIVAN, D H
PA (ARIZ) ARIZONA CHEM CORP
CYC 1
PI US 6107456 A 20000822 (200052)* 11p C09F007-00
ADT US 6107456 A CIP of US 1998-143959 19980831, US 1998-153728 19980915
PRAI US 1998-153728 19980915; US 1998-143959 19980831
IC ICM C09F007-00
ICS C11D015-00

AB US 6107456 A UPAB: 20010815
NOVELTY - New method for processing **tall oil** (1) comprises fractionating the crude **tall oil** into a volatile fraction (F1) and a residue fraction (F2). The temperature of (F2) during fractionating is 250 - 290 deg. C and (F2) includes **sterol** or **sterol esters**.
USE - For separation of **sterol** or **sterol ester** from crude **tall oil**.
ADVANTAGE - Avoids the destruction of the **sterols** as compared to the methods reported in the prior art.
DESCRIPTION OF DRAWING(S) - The Figure shows the following:
Column 101
Crude **Tall Oil** 102
Contactor Regions 103,104,105,106
Outlet 122
Dwg.1/3
FS CPI
FA AB; GI; DCN
MC CPI: D03-H01T2; **E01**; E09-D01; E10-C04L2; **E11-Q01**; E33-A03; H02-A01
TECH UPTX: 20001018
TECHNOLOGY FOCUS - CHEMICAL ENGINEERING - Preferred Process: The process involves either fractionating the crude oil feed at 300 - 310degreesC or **esterifying** the **sterols** in the crude (1) prior to fractionation. The degree of **sterol esterification** is greater than 50%. The time of separation of (F1) and (F2) is at most 1 hour and the temperature of (F2) during fractionation does not exceed 290degreesC. (F2) comprises rosin acids (at least 15%) in addition to **sterol** or **sterol esters**. The method further comprises substantially separating the **sterols** from the non-**sterol** components of (F2) by saponifying (F2) with a mixture of sodium and potassium hydroxide. The saponified (F2) is evaporated to provide a **sterol** containing fraction which is extracted with a solvent. More than 50% of the **sterol** in (1) is recovered.
ABEX
WIDER DISCLOSURE - Separation of unsaponifiable material from (1) is also disclosed. The method comprises:
(a) saponifying (1) with a mixture of sodium and potassium hydroxides to form a salts of fatty acids and/or rosin acids;
(b) evaporating the unsaponifiable material; and
(c) acidifying the unevaporated sodium and potassium salts.

EXAMPLE - Dry crude tall oil (CTO)(49,021 lbs) per hour was coursed through a feed flasher at 320degreesC. CTO exited at a temperature of 302degreesC and entered the fractionating column with different contractor regions at 246 - 277degreesC. The residue fraction that left the fractionating column had a temperature of 264degreesC and was passed through a reboiler to increase the temperature to 276degreesC. The residue fraction reentered the column and exited at a rate of 8195 lbs per hour with a yield of 16.5%. The residue fraction included beta-sitosterol (I)(11.44%) and stigmastadiene (II)(0.44%). The CTO feed contained (I)(2.44%) and (II)(0.01%). (I) in the pitch represented 78% of the material in the feed. The volatile fraction contained (13.5%) of the total (I) in the feed. This amount was shifted to the residue fraction by increasing the reflux ratio.

L58 ANSWER 9 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2000-549141 [50] WPIX
DNC C2000-163959
TI Separating a corn fiber lipid fraction comprising **phytosterol esters** and **phytosterols** useful e.g. as **hypocholesterolemics**, comprises contacting the corn fibers with a protease enzyme and solvent extraction.

DC A11 B01 B07 D13 D16 D17 **D23** E13
IN ARUMUGAM, B K; BLAIR, L; BUCHANAN, C M; BUCHANAN, N L; DEBENHAM, J S; LINGERFELT, L R; SANDERS, J K; SHELTON, M C; VISNESKI, M J; WOOD, M D
PA (EACH) EASTMAN CHEM CO
CYC 91
PI WO 2000047701 A2 20000817 (200050)* EN 187p C11B001-02
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL OA PT SD SE SL SZ TZ UG ZW
W: AE AL AM AT AU AZ BA BB BG BR BY CA CH CN CR CU CZ DE DK DM EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG UZ VN YU ZA ZW
AU 2000032281 A 20000829 (200062) C11B001-02
US 2001020091 A1 20010906 (200154) C12P019-04
EP 1155104 A2 20011121 (200176) EN C11B001-02
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT RO SE SI
US 6352845 B1 20020305 (200224) C12P019-02
US 6388069 B1 20020514 (200239) C12P019-00
ADT WO 2000047701 A2 WO 2000-US3492 20000210; AU 2000032281 A AU 2000-32281 20000210; US 2001020091 A1 Provisional US 1999-119399P 19990210, Cont of US 2000-502077 20000210, US 2001-818209 20010327; EP 1155104 A2 EP 2000-910139 20000210, WO 2000-US3492 20000210; US 6352845 B1 Provisional US 1999-119399P 19990210, US 2000-502099 20000210; US 6388069 B1 Provisional US 1999-119399P 19990210, US 2000-502077 20000210
FDT AU 2000032281 A Based on WO 200047701; EP 1155104 A2 Based on WO 200047701
PRAI US 1999-119399P 19990210; US 2000-502077 20000210; US 2001-818209 20010327; US 2000-502099 20000210
IC ICM C11B001-02; C12P019-00; C12P019-02; C12P019-04
ICS A23K001-14; C07M001-00; C08B003-00; C08B011-00; C08B037-14; C12S003-00; C13K013-00; D21C005-00
AB WO 200047701 A UPAB: 20001010
NOVELTY - Methods of separating from corn fiber, products such as corn fiber lipid fraction, animal feed, cellulose **esters**, monosaccharides and arabinoxylan and its **esters** and ethers, are new.

DETAILED DESCRIPTION - Separating a corn fiber lipid fraction (CFLF) having **phytosterol esters** (PSE) and **phytosterols** (PS) comprises:

(a) providing a mixture of corn fiber (CF) and water;

(b) contacting the mixture with a protease enzyme to give a proteolyzed CF (PCF) and a liquid;

(c) separating the liquid from the PCF; and

(d) extracting the PCF with at least one organic solvent (OS) to give a CFLF/OS solution having PSE and PS.

INDEPENDENT CLAIMS are also included for the following:

(1) a method of separating from CF a lipid fraction (LF) having PSE and PS comprising;

(a) heating an aqueous mixture of unground CF;

(b) contacting the mixture of (a) with at least one enzyme suitable for digesting starch to give a mixture of a destarched CF and a liquid comprising soluble carbohydrates;

(c) contacting the mixture of (a) or (b) with a protease enzyme to give a PCF and a liquid;

(d) separating the liquid of (c) from the CF to give a destarched, PCF; and

(e) extracting the destarched, PCF with at least one OS to give a CFLF/OS solution having PSE and PS;

(2) a method of separating a CFLF having PSE and PS comprising:

(a) providing a mixture of unground CF and water;

(b) separating the liquid from the CF to give a water wet CF; and

(c) extracting the water wet CF with at least one polar OS to give a CFLF/polar OS solution having PSE and PS;

(3) a CFLF containing PS and PSE obtained via solvent extraction of a PCF, where the concentration of PS or PSE in the LF is at least 1.4 times greater than the concentration of PS or PSE in the LF of a non PCF;

(4) a method of obtaining soluble proteins and carbohydrates from CF;

(5) a method of obtaining animal feed;

(6) a method of obtaining a cellulose material (CM) from CF;

(7) a method of obtaining a cellulose **ester** from CF comprising:

(a) heating a mixture of CF and a liquid;

(b) contacting the mixture of (a) with a protease enzyme to give a PCF and a liquid;

(c) separating the liquid from the PCF;

(d) contacting the PCF at least once with an alkaline extractant to give an ICM and a first liquid comprising arabinoxylan (I);

(e) separating (I) at at least 60 deg. C to give a CM having a cellulose content of at least 80% and consisting essentially of cellulose I;

(f) optionally subjecting the CM of (e) to at least one of a water rinsing step, an additional alkaline extractant contacting step, an alkaline H2O2 bleaching step, à xylanase enzyme contacting step, or an acid rinsing step; and

(g) contacting the CM of (e) or (f) with an O-alkylating agent to give a cellulose ether;

(8) a method of extracting (I) from CF comprising:

(a) heating an aqueous mixture of CF and a liquid;

(b) contacting the mixture of (a) with a protease enzyme to give a PCF and a liquid;

(c) separating the liquid from the PCF;

(d) contacting the PCF at least once with an alkaline extractant to give an ICM and a liquid comprising (I);

(e) separating the ICM from the liquid comprising (I); and

(f) optionally reducing the volume of the liquid comprising (I) by removing excess alkaline extractant to give a concentrated liquid comprising 10-50% solids, where the solids comprise (I);

(9) a method of extracting (I) from CF comprising:

(a) heating an aqueous mixture of CF and a liquid;

(b) contacting the mixture of (a) with a protease enzyme to give a PCF and a liquid;

(c) separating the liquid from the PCF;

(d) contacting the PCF at least once with an alkaline extractant to give an ICM and a liquid comprising (I), where the alkaline extractant does not comprises H2O2;

(e) separating the ICM from the liquid comprising (I); and

(f) reducing the volume of the liquid comprising (I) by removing excess alkaline extractant to give a concentrated liquid comprising 10-50% solids, where the solids comprise an (I);

(10) a method of preparing (I) esters from CF; and

(11) a method of obtaining at least one monosaccharide from CF comprising:

(a) heating an aqueous mixture of CF and a liquid;

(b) contacting the mixture of (a) with a protease enzyme to give a PCF and a liquid;

(c) separating the liquid from the PCF;

(d) contacting the PCF at least once with an alkaline extractant to give an ICM and a liquid comprising (I);

(e) separating the ICM from the liquid comprising (I);

(f) reducing the volume of the liquid comprising (I) by removing excess alkaline extractant to give a concentrated liquid comprising 10-50% solids, where the solids comprise (I);

(g) hydrolyzing the (I) from (f) in the presence of a catalyst and a solvent to give a mixture comprising at least one monosaccharide; and

(h) optionally separating the at least one monosaccharide from the mixture of (g).

USE - The methods can be used for the production of feedstock for the production of animal feed, chemicals and polymers. The plant sterol esters are useful as pharmaceuticals, particularly as hypocholesterolemics.

DESCRIPTION OF DRAWING(S) - The figure shows the corn fiber destarching rate where the data has been normalized relative to the total sugars present.

Dwg.0/15

FS CPI

FA AB; DCN

MC CPI: A03-A; A03-A05; A10-A; B01-D01; B04-A10; B04-B01B; B04-C02A2; B04-C02A3; B04-C02D; B04-N01; B10-A07; B14-F06; D03-G04; D05-A04; D05-C; D06-G; D10-A01; **E01**; E10-A07; **E11-Q01**

TECH UPTX: 20001010

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Methods: Obtaining a cellulose **ester** from CF comprises separating the CM from the liquid comprising (I) at least60degreesC to give a CM having a cellulose content of at least 50 (especially) 80%. When at least one monosaccharide is separated from CF utilizing SMB chromatography the process yields a concentrated liquid comprising 10-50 wt.% solids, where the solids comprise (I). The mixture is fed onto an SMB, thereby separating the at least 2 components into 2 individual eluant fractions.

Preferred Materials: The OS may be e.g. **methanol**, diethyl ether, etc.. The **esterifying** agent may be e.g. formic anhydride, acetic anhydride, etc. The O-alkylating agent may comprise ethylene oxide, ethyl bromide, sodium chloroacetate, etc. The acid catalyst may be e.g. trifluoroacetic anhydride, methane sulfonic acid, or p-toluene sulfonic acid.

TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Compound: The alkaline extractant may be e.g. NaOH, KOH, Ca(OH)2, NH4OH, CaCO3, K2CO3, Na2CO3, or LiOH. The acid catalyst is preferably H2SO4, or HCl. In the preparation of the monosaccharide, the method may further comprise contacting the L-arabinose with a solvent and a Mo(VI) catalyst at a temperature and pH to give a mixture of L-arabinose and L-ribose. The catalyst may be e.g. ammonium dimolybdate, sodium molybdate, or MoO3.

TECHNOLOGY FOCUS - POLYMERS - Preferred **Ester**: The cellulose **ester** may be e.g. cellulose formate, acetate, propionate, butyrate, formate acetate, formate propionate, formate butyrate, acetate propionate or acetate butyrate. The arabinoxylase **ester** may be e.g. (I) formate, (I) acetate, (I) propionate, (I) butyrate, (I) formate acetate, (I) formate propionate, (I) formate butyrate, (I) acetate propionate, or (I) acetate butyrate. Both the **ester** and arabinoxylase **ester** prepared from a water soluble (I) have a molecular weight of at least 300000, comprise a xylan main polymer chain with at least 2 branches, where the branches comprise groups of xylose, arabinose, galactose, glucouronic acid, 4-O-methyl glucouronic acid, or a mixture and are soluble in water at a pH of 1-14.

ABEX

EXAMPLE - A mixture of 5 g of destarched corn fiber (CF), 72 ml of deionized water, and 8 ml of 1 M sodium phosphate buffer (pH 7) was heated to 40degreesC before addition of 1 ml of Purafect 4000L (RTM, a protease enzyme). The mixture was stirred for 18 hours at 40degreesC and cooled to room temperature over 30 minutes. The sample was filtered, washed with 250 ml of deionized water, and dried at 50degreesC in vacuo to give 2.7 g of coarse fiber. Fine particles were removed from the filtrate by centrifugation. The fines were re-suspended in water, removed from the water by centrifugation, and dried at 50degreesC in vacuo to give 0.28 g of fines. The filtrate from which the fine particle solids were removed was concentrated to dryness to give a yellow solid. Results showed that treatment of destarched CF with a protease enzyme significantly lowered the % nitrogen in the remaining coarse CF to near the limit of detection. Significantly, nearly all of the protein was found in the fines and filtrate liquids. Furthermore, proteolysis of the destarched CF lowered

the % glucose while increasing the % xylose. In contrast, the unwanted fines had significantly more glucose. Collectively, this data demonstrated that treatment of destarched CF with a protease enzyme removed nearly all of the protein fraction, thus providing a cleaner CF that ultimately led to a cleaner arabinoxylan (I) and cellulose fraction.

L58 ANSWER 10 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN **2000-499911** [45] WPIX
DNC **C2000-150167**
TI Isolation of **phytosterols** following methyl **esterification** of **rape seed** or **soya fatty acids** comprises crystallization from 25 - 75 wt. % **methanol**, useful as **hypocholesterolemic** agents in drugs and foods.
DC B01 D13 E15
IN ARMENGAUD, R; **GUTSCHE, B**; JORDAN, V; MUSHOLT, M; **SCHWARZER, J**; SICRE, C
PA (COGN-N) **COGNIS DEUT GMBH**
CYC 24
PI DE 19916034 C1 20000803 (200045)* 3p C07J009-00 <--
WO 2000061603 A1 20001019 (200054) DE C07J009-00 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: AU CA JP NZ US
AU 2000045411 A 20001114 (200108) C07J009-00 <--
EP 1169335 A1 20020109 (200205) DE C07J009-00 <--
R: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
ADT DE 19916034 C1 DE 1999-19916034 19990409; WO 2000061603 A1 WO 2000-EP2849 20000331; AU 2000045411 A AU 2000-45411 20000331; EP 1169335 A1 EP 2000-926783 20000331, WO 2000-EP2849 20000331
FDT AU 2000045411 A Based on WO 200061603; EP 1169335 A1 Based on WO 200061603
PRAI DE 1999-19916034 19990409
IC ICM **C07J009-00**
ICS C11B013-00; C11B013-02
AB DE 19916034 C UPAB: 20000918
NOVELTY - **Phytosterols** are isolated from the fraction obtained by methyl **esterification** of **rape seed** or **soya fatty acids** by crystallization with 25 - 75 wt. % **methanol**, followed by filtration, washing and drying.
ACTIVITY - Antilipemic.
MECHANISM OF ACTION - **Cholesterol** antagonist.
USE - **Phytosterols** are useful as **hypocholesterolemic** agents in drugs or as part of food e.g. margarine, frying **oils**, sausages and ice-creams.
ADVANTAGE - The use of certain levels of **methanol** results in higher yields, due to an increase in crystallization temperature which rises in direct proportion to the amount of **methanol** used (reaching a maximum at a **methanol** content of 75 wt. % with respect to **sterol**, after which it falls off again). This allows production of **sterols** from **plant oils** containing only a small amount of **sterol**.
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B01-D02; B10-E04D; B11-B; B14-D02A2; B14-F06; D03-C02; **E01**; E10-E04L1; **E11-Q01**
TECH UPTX: 20000918
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Material: The **phytosterol** is obtained from **rape seed** or **soya**. After crystallization, the crude **sterol** is washed with a **fatty acid ester**.
ABEX
ADMINISTRATION - No dose or route of administration is given.

EXAMPLE - A rape seed oil methyl ester fraction (which contained 30 wt. % methanol) was cooled from a temperature of 100 degreesC to 10 degreesC. Sterol crystals started to come out of solution at 78 degreesC. The crystals were filtered, washed with methanol to remove any methyl ester and dried. The yield of sterol was 92 wt. %, based on the amount of sterol in the initial esterification product. A methyl ester fraction containing 100 wt. % methanol gave a yield of 78 wt. % and started to crystallize at 68 degreesC.

L58 ANSWER 11 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN **2000-431268** [37] WPIX
DNC **C2000-131045**
TI Method for the isolation of **sterols** from sulfate pulping process **tall oil** pitch..
DC B01 D13 E14 F09
IN CUFF, T J; PARKER, J E; ROBINSON, P L
PA (WEVC) WESTVACO CORP
CYC 79
PI WO 2000034305 A1 20000615 (200037)* EN 29p C07J009-00 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE GH GM HR HU ID IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZW
AU 9949585 A 20000626 (200045) C07J009-00 <--
ADT WO 2000034305 A1 WO 1999-US14137 19990712; AU 9949585 A AU 1999-49585 19990712
FDT AU 9949585 A Based on WO 200034305
PRAI US 1998-206228 19981207
IC ICM **C07J009-00**
ICS C11B013-00
AB WO 200034305 A UPAB: 20000807
NOVELTY - Method for the isolation of **sterols** from sulfate pulping process **tall oil** pitch is new.
DETAILED DESCRIPTION - Method for the isolation of **sterols** from sulfate pulping process **tall oil** pitch comprises:
(1) separating a **sterol**-rich fraction from a saponified **tall oil** pitch;
(2) blending a hydrocarbon solution of the **sterol**-rich fraction with a first wash **alcohol** solvent at a temperature greater than the crystallization temperature of the **sterol** to produce a hydrocarbon/**sterols**/**alcohol** solution;
(3) adding a first wash water to the hydrocarbon/**sterols**/**alcohol** solution to give a first upper hydrocarbon phase and a **lower alcohol**/water phase;
(4) removing the first **lower alcohol**/water phase;
(5) adding water to the hydrocarbon phase at a temperature higher than the hydrocarbon phase to produce a second upper hydrocarbon phase and a **lower** water phase;
(6) removing the **lower** water phase;
(7) allowing the remaining hydrocarbon phase to cool to about 20-40 deg. C to produce **sterol** crystals; and
(8) recovering the **sterol** crystals from the cooled solution.
USE - The **sterols** are useful as a dietary supplement in foods to reduce **cholesterol** levels in humans.
Dwg.0/1
FS CPI
FA AB; DCN
MC CPI: B04-J02; B11-B; B14-F06; D03-H01T2; **E01**; **E11-Q01**; F05-A02C
TECH UPTX: 20000807
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: The extraction

neutrals of **tall oil** pitch are derived by a process selected from solvent extraction and **distillation**. The temperature in step (b) is at least 25 degreesC, preferably at least 60 degreesC and the temperature in step (e) is at least 60 degreesC. The method further comprises washing the recovered **sterol** crystals of step (h) with a hydrocarbon solvent to obtain a high yield (at least 60%, preferably at least 65%, especially at least 70%) of **sterols** of high purity (at least 92%, preferably at least 95%). Steps (a) and (b) are combined by the first wash alcohol with the first wash water. Preferred Components: The **sterol**-rich fraction is a neutrals fraction derived by solvent extraction. The hydrocarbon solvent is a 5-12C hydrocarbon selected from pentane, hexane, heptane, iso-octane and mixtures of these. The alcohol solvent is an aliphatic alcohol selected from **methanol, ethanol, butanol**, iso-**propanol** and mixtures of these.

ABEX

EXAMPLE - In a first quarter fractional factorial, Plackett-Burman designed experiment for the dual decantation crystallization process, 16 experimental runs were conducted with six center points giving a total of 22 randomized runs. Both wash steps (the first was done with alcohol and water combined) were done at 60-65 degreesC. Yield and purity values ranged from 28-74% and 77-94% respectively.

L58 ANSWER 12 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN **2000-413798** [36] WPIX
DNC **C2000-125516**
TI Purification of **phytosterols** useful as **hypocholesterolemic** agents and as food additives comprises crystallization from a saturated hydrocarbon solvent.
DC B01 D13 E15
IN **GUTSCHE, B; SCHWARZER, J**
PA (COGN-N) **COGNIS DEUT GMBH**
CYC 24
PI DE 19906551 C1 20000629 (200036)* 3p C07J009-00 <--
WO 2000047570 A1 20000817 (200041) DE C07D311-72
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: AU CA JP NZ US
AU 2000029066 A 20000829 (200062) C07D311-72
EP 1150968 A1 20011107 (200168) DE C07D311-72
R: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
ADT DE 19906551 C1 DE 1999-19906551 19990213; WO 2000047570 A1 WO 2000-EP903 20000204; AU 2000029066 A AU 2000-29066 20000204; EP 1150968 A1 EP 2000-907499 20000204, WO 2000-EP903 20000204
FDT AU 2000029066 A Based on WO 200047570; EP 1150968 A1 Based on WO 200047570
PRAI DE 1999-19906551 19990213
IC ICM C07D311-72; **C07J009-00**
ICS C07J075-00
AB DE 19906551 C UPAB: 20000807
NOVELTY - **Phytosterols** obtained by alkali-catalysed **esterification** of the residue obtained from the production of e.g. **sunflower fatty acid** methyl **ester** are purified by crystallization using a saturated 5-10C hydrocarbon solvent, at a temperature where the **ester** is normally liquid.
DETAILED DESCRIPTION - Preparation of **phytosterols** comprises alkali-catalysed **esterification** of the residue obtained from the production of methyl **esters** from **methanol**, followed by neutralization of the catalyst and separation of any unreacted alcohol. The **phytosterol** is crystallized out by reducing the temperature, followed by filtration, washing and drying.
ACTIVITY - Antilipemic.
MECHANISM OF ACTION - **Cholesterol** antagonist.
USE - **Phytosterols** are useful as

hypocholesterolemic agents and as additives for food e.g. margarine, frying **oils**, sausages and ice-creams.

ADVANTAGE - The technique described in the invention results in high yields of a product which is virtually free of citrostadienol.

Dwg.0/0

FS CPI

FA AB; DCN

MC CPI: B01-D02; B04-D03; B14-F06; B14-L06; D03-A01; D03-C01; D03-C02; D03-E08; D03-H01T2; **E01**; **E11-Q01**

TECH UPTX: 20000801

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: The residue is obtained from **sunflower fatty acid** methyl **ester** or **tall oil** pitch production.

Dissolution takes place at 60-80 degrees centigrade, and the solvent is selected from hexane and/or heptane. During the crystallization, a 1-25 wt. % aqueous **methanol** solution is added; the solution makes up 1-15 wt. % of the hydrocarbon. The **phytosterol** contains less than 0.5 wt. % citrostadienol.

ABEX

ADMINISTRATION - No dose or route of administration is given.

EXAMPLE - A distillation residue (200 g) obtained from the production of sunflower fatty acid methyl ester was mixed with 78 g methanol. (The residue contained 15 wt. % glycerides and 28 wt. % free or bound sterol.) The mixture was reacted with 3.8 g, 30 wt. % sodium methylate solution and stirred at 70 degrees centigrade for four hours. The catalyst was neutralized by adding citric acid (4.2 g dissolved in 19 g methanol). Any unreacted methanol was distilled off and the residue was washed with water at 65 degrees centigrade. The crude product was mixed with 400 g hexane, 26 g methanol and 8 g water, and cooled to 20 degrees centigrade. The mother liquor was filtered off and the residue was dried to give 41 g sterol, which was free of citrostadienol. In a comparison study, the esterification product was mixed with methanol in a 1:1 weight ratio, followed by filtration, washing with aqueous methanol and drying. Further purification of the product (100 g) by dissolution in heptane at 70 degrees centigrade, mixing with 20 g methanol and cooling to 20 degrees centigrade gave 75 g of a sterol containing 4.2 % citrostadienol.

L58 ANSWER 13 OF 34 WPIX (C) 2002 THOMSON DERWENT

AN **2000-349613** [30] WPIX

DNC **C2000-106287**

TI Isolation of **sterols** from sulphate pulping process **tall oil** pitch comprises, saponification, extraction, partition and crystallisation.

DC B01 D13 E15

IN CUFF, T J; PARKER, J E; ROBINSON, P L

PA (WEVC) WESTVACO CORP

CYC 79

PI US 6057462 A 20000502 (200030)* 7p C07J009-00 <--

WO 2000027867 A1 20000518 (200032) EN C07J009-00 <--

RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE

W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE GH GM HR HU ID IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZW

AU 9949584 A 20000529 (200041) C07J009-00 <--

ADT US 6057462 A US 1998-187448 19981106; WO 2000027867 A1 WO 1999-US14136 19990712; AU 9949584 A AU 1999-49584 19990712

FDT AU 9949584 A Based on WO 200027867

PRAI US 1998-187448 19981106

IC ICM **C07J009-00**

ICS C11B013-00

AB US 6057462 A UPAB: 20000624

NOVELTY - Isolation of **sterols** from sulphate pulping process **tall oil** pitch.
DETAILED DESCRIPTION - The method comprises:
(a) saponification of the **tall oil** pitch;
(b) extraction of the neutral fraction;
(c) mixing a hydrocarbon solution of the neutrals with an alcohol at a temperature above the crystallisation temperature of the **sterol**;
(d) adding water;
(e) removing the alcohol/water phase;
(f) cooling the organic phase to 20 to 30 deg. C;
(g) recovering the crystallized **sterol**; and
(h) washing the **sterol** with a hydrocarbon solvent.
USE - The method is useful for obtaining **sterols** from crude **tall oil** recovered from the black liquor residue of wood pulping processes.
ADVANTAGE - The process gives at least 60% recovery of **sterols** with a purity of at least 90%.
Dwg.0/2

FS CPI
FA AB; DCN
MC CPI: B10-C04D; B10-C04E; D03-C01; **E01**; **E11-Q01**
TECH UPTX: 20000624
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: The extraction neutrals are preferably derived from solvent extraction or **distillation**. The hydrocarbon solvent is preferably a 5-10C hydrocarbon, especially pentane, hexane, heptane or iso-octane. The alcohol is preferably an aliphatic alcohol, especially **methanol**, **ethanol**, **butanol** or 2-**propanol**. Step (a) is preferably carried out at above 70 degrees C.
ABEX
EXAMPLE - Isolation of sterols comprised dissolving the extract in 2 parts of hexane, adding 0.55 parts methanol, 0.5 parts water, cooling to 30 degrees C and crystallising for 1 hour. This gave a 65.9% sterol yield with a purity of 94.3% and no wax alcohol.

L58 ANSWER 14 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2000-271374 [23] WPIX
DNC C2000-082866
TI New method for separating a **sterol** or **sterol ester** from crude **tall oil** comprises fractionating the crude **oil** into a residue fraction and a volatile fraction..
DC B01 **D23** E19
IN HUIBERS, D T A; ROBBINS, A M; SULLIVAN, D H
PA (UNBC) UNION CAMP CORP
CYC 79
PI WO 2000015652 A1 20000323 (200023)* EN 35p C07J009-00 <--
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL OA PT SD SE SZ UG ZW
W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE HU ID IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UZ VN
AU 9895691 A 20000403 (200034) C07J009-00 <--
ADT WO 2000015652 A1 WO 1998-US19271 19980915; AU 9895691 A AU 1998-95691 19980915, WO 1998-US19271 19980915
FDT AU 9895691 A Based on WO 200015652
PRAI WO 1998-US19271 19980915
IC ICM **C07J009-00**
AB WO 200015652 A UPAB: 20000516
NOVELTY - A new method for separating a **sterol** or **sterol ester** from crude **tall oil** comprises fractionating the crude **oil** into a residue fraction and a

volatile fraction. The temperature of the residue fraction during fractionation does not exceed 290 deg. C and the residue fraction includes the **sterol** or **sterol ester**.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for a method for separating unsaponifiable material from a **tall oil** stream comprising saponifying the stream with a mixture of sodium hydroxide and potassium hydroxide to form sodium and potassium salts of **fatty acids**, rosin **acids** or both, evaporating the unsaponifiable material and acidulating the unevaporated sodium and potassium salts.

USE - The method is useful for separating **sterols** and **sterol esters** (e.g. **sitosterol**, **stigmasterol**, **campesterol**, sitostanol, and campestanol) from crude **tall oil**. The method is particularly applicable to the recovery of beta -**sitosterol** which is indicated as an agent for reducing circulating **cholesterol** levels.

DESCRIPTION OF DRAWING(S) - The drawing shows a schematic view of a pitch-collecting apparatus.

Feed flasher 107

Column 101

Contactors 104,105

Reboiler 118

Outlet 122

Dwg.1/3

FS CPI

FA AB; GI; DCN

MC CPI: B01-D02; B04-J02; B14-D02A2; **D10-A04**; **E01**; **E11-Q01**

TECH UPTX: 20000516

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred method: The temperature of the residue fraction is about 250-270 degreesC. Fractionation is carried out in a thin film evaporator at a temperature below about 250 degreesC. The temperature of the crude **tall oil** fractionation feed is about 300-310 degreesC. The time of separating the residue fraction from the volatile fraction does not exceed about 1 hour after which the residue fraction is allowed to cool. The residue fraction comprises at least about 15% rosin acids. More than 50% of the **sterols** in the crude **tall oil** is recovered.

The method further comprises **esterification** of the **sterols** in the crude **tall oil** prior to or during fractionation, the degree of **esterification** being greater than 50%. The method further comprises separating the **sterols** from the non-**sterol** components of the residue fraction. The method further comprises saponifying the residue fraction to recover free **sterols**, the saponification being carried out by combining the residue fraction with a mixture of sodium hydroxide and potassium hydroxide. The saponified residue fraction is subjected to evaporation to provide an evaporated fraction containing **sterols**. The saponified residue fraction is extracted with a solvent to provide an extract containing **sterols**. The residue fraction comprises rosin acids (at least about 15%). Preferred components: The **tall oil** stream from which unsaponifiable material is separated is a **tall oil** pitch stream.

ABEX

EXAMPLE - Figure 1 illustrates a pitch-collecting apparatus. Crude tall oil (49021 pounds/hour) was coursed through feed flasher (107) set at 320 degreesC. The crude tall oil exited at a temperature of 302 degreesC and entered column (101) via tube (110). The crude tall oil fractionated via interaction of the contactor regions and the heated oil, contactor (104) had a temperature range of 246-250 C and contactor (105) had a temperature range of 272-277 degreesC. The residue fraction left the column via tube (123) which had a temperature of 264 degreesC and coursed through the

reboiler (118), attaining a temperature of 276 degreesC as it exited and re-entered the column (101) via tube (124). The residue fraction was removed from column (101) at outlet (122). The residue fraction included beta-sitosterol (11.44%) and stigmastadiene (0.44%).

L58 ANSWER 15 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 2000-161097 [14] WPIX
DNC C2000-050427
TI Isolation of **sterols** free of dimer **fatty acids** from crude **tall oil** for use as dietary supplement in foods to reduce **cholesterol** levels in humans.
DC D13 **D23** E15 F09
IN BYRNE, J F; CUFF, T J; ROBINSON, P L; YAN, Z Q
PA (WEVC) WESTVACO CORP
CYC 79
PI WO 2000004039 A1 20000127 (200014)* EN 19p C07J009-00 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE GH GM HR HU ID IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZW
AU 9949587 A 20000207 (200029) C07J009-00 <--
ADT WO 2000004039 A1 WO 1999-US14139 19990712; AU 9949587 A AU 1999-49587 19990712
FDT AU 9949587 A Based on WO 200004039
PRAI US 1998-115002 19980714
IC ICM **C07J009-00**
ICS C11B013-00
AB WO 200004039 A UPAB: 20000320
NOVELTY - **Sterol** isolation from a hydrocarbon solution of **tall oil** pitch neutrals is as follows:
(a) solution is blended with 1-4:0.1-0.5 mixture of alcohol and water respectively, at about 40 - 50 deg. C.
(b) the solution phase is isolated and cooled to about 20 - 30 deg. C and
(c) equal part of water is added to it with agitation to precipitate the **sterol** granules, which are recovered by filtration.
USE - As dietary supplement in foods to reduce **cholesterol** levels in humans.
ADVANTAGE - The single decantation precipitation process gives **sterol** of higher purity and in higher yield when compared to the prior art. The impurities dimer **fatty acids** are removed.
Dwg.0/1
FS CPI
FA AB; DCN
MC CPI: D03-H01T; D10-A02; **E01**; **E11-Q01**; F05-A02C
TECH UPTX: 20000320
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: Dry neutrals are dissolved in a hydrocarbon solvent in a ratio of 1:2-5 respectively. The neutrals are obtained by solvent extraction and **distillation**. The hydrocarbon solvent is 5-10C straight- or branched-chain hydrocarbon and the alcohol solvent is an aliphatic alcohol.
ABEX
SPECIFIC COMPOUNDS - The hydrocarbon solvent is pentane, hexane, heptane or isooctane. The alcohol is methanol, ethanol, propanol, butanol or isopropanol.

EXAMPLE - Concentrated neutrals was dissolved in heptane in 1:2-5 ratio respectively. To this, methanol and water were added at 40-50degreesC. The sterol-rich heptane phase was isolated and cooled. Water was added with agitation and the precipitated solid sterols, which were then filtered. It was found that the reference RSV 7253-85 gave yield of 70.8% and the

sterol purity of 97.7%. Another reference RSV 7253-71B had the sterol purity of 96.2% and the yield of 78.8%.

L58 ANSWER 16 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN **2000-161096** [14] WPIX
DNC **C2000-050426**
TI Isolation of **sterols** free of wax alcohols from crude **tall oil** for use as dietary supplement in foods to reduce **cholesterol** levels in humans.
DC D13 E15 F09
IN BYRNE, J F; CUFF, T J; ROBINSON, P L; VICENTE, R S
PA (WEVC) WESTVACO CORP
CYC 79
PI WO 2000004038 A1 20000127 (200014)* EN 19p C07J009-00 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE GH GM HR HU ID IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZW
AU 9949582 A 20000207 (200029) C07J009-00 <--
ADT WO 2000004038 A1 WO 1999-US14134 19990712; AU 9949582 A AU 1999-49582 19990712
FDT AU 9949582 A Based on WO 200004038
PRAI US 1998-115003 19980714
IC ICM **C07J009-00**
ICS C11B013-00
AB WO 200004038 A UPAB: 20000320
NOVELTY - Isolation of **sterols** from a hydrocarbon solution of **tall oil** pitch neutrals is as follows:
(a) solution is blended with 1-4:0.1-05 of alcohol and water respectively at 50-60 deg. C,
(b) cooled to a final crystallization temperature of 20-40 deg. C and
(c) equal part of water is added to it with agitation to precipitate the **sterol** granules which are recovered by filtration.
USE - As dietary supplement in foods to reduce **cholesterol** levels in humans.
ADVANTAGE - The direct precipitation process gives a **sterol** of higher purity and higher yield when compared to the prior art. Impurities like wax alcohols are removed.
Dwg.0/1
FS CPI
FA AB; DCN
MC CPI: D03-H01T2; **E01**; **E11-Q01**; F05-A02C
TECH UPTX: 20000320
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Method: The hydrocarbon solution of the neutrals is made by dissolving dry neutrals in a hydrocarbon solvent in 1:2-5 ratio respectively by heating. The neutrals are obtained by solvent extraction and **distillation**. The hydrocarbon solvent is 5-10C straight or branched hydrocarbon and the solvent is an aliphatic alcohol.
ABEX
SPECIFIC COMPOUNDS - The hydrocarbon solvent is pentane, hexane, heptane or iso-octane. The alcohol is methanol, ethanol, butanol or iso-propanol.

EXAMPLE - Dry neutrals were obtained from a simple solvent extraction of the tall oil pitch. They were dissolved in a refluxing mixture of heptane and methanol in a ratio of 2 - 5:0.2 - 0.5 parts. The mixture was cooled at 20-35degreesC and 0.5-1.5 parts water added with agitation to precipitate the sterols and filtered. The solids obtained were washed with 2 parts heptane, dried, weighed and analyzed for sterol purity. The sterol obtained from the process in which parts of heptane, methanol and wash water were 2, 0.2 and 0.5 respectively at 30degreesC crystallization temperature and 20.9 rpm agitation time gave 87.5% purity and the best

yield of 82.9%.

L58 ANSWER 17 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1999-542697 [46] WPIX
DNC C1999-158527
TI Use of residue of crude **fatty acids** for the manufacture of feed for crustaceans.
DC D13 **D23** E17 E19
IN FITIE, A F; FILIPPUS, A
PA (CROY-N) CROY ASSOC BV
CYC 29
PI EP 945071 A1 19990929 (199946)* EN 7p A23K001-16
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT RO SE SI
NL 1008684 C2 19990927 (200002) A23K001-165
CN 1230355 A 19991006 (200006) A23K001-18
JP 11318345 A 19991124 (200006) 4p A23K001-06
US 6136368 A 20001024 (200055) A23J007-00
MX 9902679 A1 20000901 (200139) A23K001-18
ADT EP 945071 A1 EP 1999-200785 19990311; NL 1008684 C2 NL 1998-1008684 19980324; CN 1230355 A CN 1999-103033 19990322; JP 11318345 A JP 1999-78046 19990323; US 6136368 A US 1999-274170 19990323; MX 9902679 A1 MX 1999-2679 19990322
PRAI NL 1998-1008684 19980324
IC ICM A23J007-00; A23K001-06; A23K001-16; A23K001-165; A23K001-18
ICS A23K001-10; A23K001-175; C11C001-04; C11C001-10
AB EP 945071 A UPAB: 19991110
NOVELTY - Use of feed for crustaceans of a **residue** in the **distillation** of crude **fatty acids**, the **fatty acids** are obtained from the hydrolysis of natural, chiefly animal fats.
USE - The process is useful for in the manufacture of feeds for crustaceans.
ADVANTAGE - The feed is useful in the cultivating of crustaceans as the natural feed available is not sufficient to support good growth in crustaceans and overcomes the problems associated with expensive feeds.
DESCRIPTION OF DRAWING(S) - The drawing diagrammatically shows a **distillation** process in which crude **fatty acids** are produced from natural fats and oils. The **distillation** takes place in a **distillation** tower (13).
Dwg.1/3
FS CPI
FA AB; GI; DCN
MC CPI: D03-G03; D10-A02; **E01**; E05-G09D; E10-C04K; E10-C04L; **E11-Q01**; E11-Q02
TECH UPTX: 19991110
TECHNOLOGY FOCUS - FOOD - Preferred Feed: A protein carrier and/or silicate is preferably added to the residue especially **sterols** e.g. soy lecithin.

L58 ANSWER 18 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1999-518581 [43] WPIX
DNC C1999-151448
TI Method for the preparation of **phytosterol** compounds from **tall oil** pitch containing steryl **esters**.
DC **D23** E15 F09
IN MACMILLAN, A K; NORMAN, H S O; WONG, A
PA (BCCH-N) BC CHEM LTD
CYC 85
PI WO 9942471 A1 19990826 (199943)* EN 23p C07J009-00 <--
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL OA PT SD SE SZ UG ZW
W: AL AM AT AU AZ BA BB BG BR BY CH CN CU CZ DE DK EE ES FI GB GD GE

GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD
MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA
UG US UZ VN YU ZW

AU 9926057 A 19990906 (200003) C07J009-00 <--
CA 2230373 A1 19990820 (200005) EN C07J009-00 <--
EP 1056767 A1 20001206 (200064) EN C07J009-00 <--
R: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
JP 2002504489 W 20020212 (200215) 32p C07J009-00 <--

ADT WO 9942471 A1 WO 1999-CA150 19990219; AU 9926057 A AU 1999-26057 19990219; CA 2230373 A1 CA 1998-2230373 19980220; EP 1056767 A1 EP 1999-905995 19990219, WO 1999-CA150 19990219; JP 2002504489 W WO 1999-CA150 19990219, JP 2000-532423 19990219

FDT AU 9926057 A Based on WO 9942471; EP 1056767 A1 Based on WO 9942471; JP 2002504489 W Based on WO 9942471

PRAI CA 1998-2230373 19980220

IC ICM **C07J009-00**
ICS C11B013-00

AB WO 9942471 A UPAB: 19991020
NOVELTY - A method for the preparation of **phytosterol** compounds from **tall oil** pitch containing steryl **esters** comprises use of **distillation** techniques to isolate a **phytosterol** concentrate that can by crystallisation yield a high purity product.

DETAILED DESCRIPTION - A method of preparing **phytosterols** from **tall oil** pitch containing steryl **esters** comprises:

(a) converting the steryl **esters** to free **phytosterols** while in the pitch to produce a modified pitch containing the free **phytosterols**;

(b) removing light ends from the modified pitch by evaporation to produce a bottom fraction containing the free **phytosterols**;

(c) evaporating the bottom fraction to produce a light phase **distillate** containing the free **phytosterols**;

(d) dissolving the light phase **distillate** in a solvent comprising an alcohol to produce a solution containing the free **phytosterols**;

(e) cooling the solution to produce a slurry with the free **phytosterols** crystallised in the slurry; and

(f) washing and filtering the slurry to isolate the crystallised **phytosterols**.

USE - None given.

ADVANTAGE - The method produces high purity **phytosterol** crystals from **tall oil** pitch.

Dwg.0/1

FS CPI
FA AB; DCN
MC CPI: D10-A02; **D10-A04**; **E01**; **E11-Q01**; F05-A02B

TECH UPTX: 19991020
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Materials: The solvent comprises: a low molecular weight monohydric alcohol, preferably **methanol**, **ethanol**, 2-**propanol**, or a combination of these; and optionally water. A like solvent may be used for washing and filtering the slurry.
Preferred Method: Step (a) comprises the steps of: saponifying the **tall oil** pitch with an alkali metal base; neutralising the saponified pitch with an acid; and heating the neutralised pitch to remove water, the resulting modified pitch preferably comprising less than 1 wt.% water. The light ends are removed in a wiped film evaporator operating at 0.1-10 millibars and 160-280 degreesC; the bottom fraction produced is evaporated in a wiped film evaporator operating at 0.01-1.0 millibars and 180-300 degreesC. The method may further comprise evaporating the light phase **distillate** after step (c) and before

step (d) to enhance the concentration of free **phytosterols**, crystallisation of **phytosterols** is effected at 0-35 degreesC.
TECHNOLOGY FOCUS - INORGANIC CHEMISTRY - Preferred Alkali Metal Base: The alkali metal base is NaOH, KOH, and mixtures of NaOH and KOH. The alkali metal base to **tall oil** pitch is 1-15 wt%.
Preferred Acid: The acid is H2SO4, HCl, H3PO4 and a combination of two or more of the above acids.
Preferred Conditions: The saponification is conducted at 100-250 degreesC for 60-300 minutes. The neutralization is at 10-100 degreesC for 1-10 hours.

ABEX

EXAMPLE - A sample of tall oil pitch was saponified, neutralised, and dewatered to produce a modified pitch containing 141 mg free phytosterols/g. The modified pitch was fed into a series of 0.1 m2 wiped evaporators for a 2-stage evaporation, the distillate from the first stage (5.94 mbar, 225 degreesC) being fed to the second stage (0.32 mbar, 251 degreesC). The second distillate, containing 248 mg free phytosterols/g, was dissolved in methanol at 65 degreesC, wt. ratio solvent to distillate 1.5 : 1.0. The solution was cooled to 30-35 degreesC, and the phytosterol crystals filtered, washed with methanol, and dried. Crystal purity was 983 mg pure phytosterols/g dry cake; crystal yield was 41.9 % based on phytosterols in the distillate.

L58 ANSWER 19 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1999-494514 [41] WPIX
DNC C1999-145026
TI Isolating purified **tall oil sterol** from natural **tall oil** components for use in pharmaceuticals, cosmetics and processing materials.
DC B01 B04 D21 **D23** E15
IN ABE, T; EZAKI, Y
PA (ARAK) ARAKAWA CHEM IND LTD
CYC 21
PI WO 9941272 A1 19990819 (199941)* JA 21p C07J009-00 <--
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE
W: CA US
JP 11228593 A 19990824 (199944) 4p C07J001-00
EP 990662 A1 20000405 (200021) EN C07J009-00 <--
R: FI FR
ADT WO 9941272 A1 WO 1999-JP619 19990212; JP 11228593 A JP 1998-52958 19980217; EP 990662 A1 EP 1999-902894 19990212, WO 1999-JP619 19990212
FDT EP 990662 A1 Based on WO 9941272
PRAI JP 1998-52958 19980217
IC ICM C07J001-00; **C07J009-00**
ICS C07J075-00
AB WO 9941272 A UPAB: 19991011
NOVELTY - Isolating purified **tall oil sterol** from natural **tall oil** components comprises a step of contacting the natural **tall oil** components with **lower alcohol** at temperature exceeding the boiling point at ordinary pressure of the solvent.
DETAILED DESCRIPTION - Isolating purified **tall oil sterol** from natural **tall oil** components comprises:
(a) contacting the natural **tall oil** components with **methanol**, aqueous **methanol**, aqueous **ethanol**, aqueous isopropanol and/or aqueous n-**propanol** at a temperature exceeding the boiling point at ordinary pressure of the solvent;
(b) removing the undissolved material; and
(c) crystallizing the **tall oil sterol** from the solution and recovering the crystals by filtration.
USE - For isolating purified **tall oil**

sterol, useful in pharmaceuticals, cosmetics and processing materials, from natural **tall oil** components.

ADVANTAGE - High purity **tall oil sterol** is readily obtained and easily isolated.

Dwg.0/0

FS CPI

FA AB; DCN

MC CPI: B04-B01C1; B04-J01; D08-B; D10-A02; **E01**; **E11-Q01**

TECH UPTX: 19991105

TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Process: **Methanol** is used in an amount of 100-150 wt.% of the natural **tall oil** components and the mixture is contacted at 70-170degreesC. The crystallization is at greater than 50degreesC.

ABEX

EXAMPLE - Tall oil components containing 45 wt.% tall oil sterol (100 g) in methanol (300 g) were heated 90degreesC under pressure and the mixture was stirred under the same conditions for 30 minutes. The pressure was released and after 5 minutes the liquid was decanted to leave the solids. The liquid was allowed to stand at 65degreesC to allow crystals to form. The crystals were removed by filtration, washed with methanol (30 g) and dried to give 21 g (96 wt.%) of the tall oil sterol.

L58 ANSWER 20 OF 34 WPIX (C) 2002 THOMSON DERWENT

AN 1999-419588 [36] WPIX

CR 1999-419598 [31]

DNC C1999-123489

TI Extraction of **sterol** and tocol useful for pharmaceuticals, nutrient compositions, cosmetics and personal care products..

DC B01 B02 D21 **D23** E13 E15

IN HETHERINGTON, M A

PA (FYTO-N) FYTOKEM PROD INC

CYC 1

PI CA 2213112 A 19990214 (199936)* 13p C07J009-00 <--

ADT CA 2213112 A CA 1997-2213112 19970814

PRAI CA 1997-2213112 19970814

IC ICM **C07J009-00**

ICS C07D311-72

AB CA 2213112 A UPAB: 19990908

NOVELTY - A process for extraction of lipids from an unsaponifiable **plant** material comprises sequentially extracting two lipids by dissolving an unsaponifiable **plant** material in an atropic solvent.

DETAILED DESCRIPTION - A process for sequentially extracting two lipids from unsaponifiable **plant** material comprises:

(1) extracting a first lipid by:

(a) dissolving an unsaponifiable **plant** material in an atropic solvent to form a solution (I);

(b) cooling (I) until a precipitate of the first lipid (II) is formed, leaving a lipid supernatant (III); and

(c) recovering (II); then

(2) extracting a second lipid by:

(a) **distilling** (III) to obtain a **residue** of the second lipid (IV); and

(b) drying (IV) to remove traces of the atropic solvent.

USE - For extracting lipids from **plants** in the manufacture of pharmaceuticals, nutrient compositions, cosmetics and personal care products.

ADVANTAGE - The process may be applied to all types of unsaponifiable **plant** material produced by refining of **vegetable oil**. The method can be used on a small (e.g. laboratory) to large manufacturing scale and is compatible with standard operations and equipment currently used for processing and refining oilseeds and other **plant** materials. The yield of the process, as well as the

homogeneity and physical appearance of the **sterol** are improved.
The process allows recycling of the solvent and energy.
DESCRIPTION OF DRAWING(S) - The schematic diagram shows the extracting process.
Dwg.2/2/1

FS CPI
FA AB; GI; DCN
MC CPI: B01-D02; B03-H; D08-B; D10-A01; **E01**; E06-A01; **E11-Q01**
TECH UPTX: 19990908
TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Products: The first lipid extracted is a **sterol**, especially **phytosterol**, and the second lipid is tocol, especially alpha and gamma tocols (common forms of vitamin E).
Preferred Solvents: The atropic solvent is pentane, isopentane, isohexane, heptane, octane or preferably hexane.
Preferred Plant Materials: The plant material, especially deodorizer **distillate**, is derived from **rapeseed**, corn, **sunflower**, **palm**, **soybean** or preferably canola.
Preferred Process: The plant material is preferably dissolved in a hot atropic solvent. The process comprises an additional step for recycling the warm atropic solvent by reusing it to dissolve the plant material. The different steps of the process can be synchronized to allow continuous operability.
ABEX
SPECIFIC COMPOUNDS - The extracted lipids are sterol and tocol.

EXAMPLE - Deodorizer distillate (2.4 g) from canola was dissolved in hexane (20 ml) heated to 60 degrees C. The solution was refrigerated for 48 hours then filtered, washed with a little cold hexane and evaporated by air for 12 hours. White crystalline solid of phytosterols deposited on the inner surfaces of the vessel. The cold solution was drained and decanted into a second vessel. The solvent was boiled away and the resulting residue evaporated under low vacuum. The resulting phytosterol fraction had over 99% purity. The phytosterol was white, in 60-80 % yield and had low odor. The phytosterol fraction comprised beta-sitosterol (45%), brassicasterol (30%), campesterol (20%) and stigmasterol (5%).

L58 ANSWER 21 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1998-159079 [14] WPIX
DNC C1998-051235
TI Oryzanol concentrate separation - from oryzanol-containing fatty substance, particularly crude oil, in highly economical process.
DC B01 D13 D21 **D23** E14
IN HOFMAN, C; ZWANENBURG, A; VAN AMERONGEN, M P; ZWANEMBURG, A
PA (UNIL) UNILEVER PLC; (UNIL) UNILEVER NV; (LIPT-N) LIPTON DIV CONOPCO INC
CYC 79
PI WO 9801519 A1 19980115 (199814)* EN 28p C11B003-00
RW: AT BE CH DE DK EA ES FI FR GB GH GR IE IT KE LS LU MC MW NL OA PT SD SE SZ UG ZW
W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE GH HU IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZW
AU 9735400 A 19980202 (199826) C11B003-00
ZA 9705992 A 19990331 (199918) 24p C11B000-00
EP 912665 A1 19990506 (199922) EN C11B003-00
R: BE DE DK ES FI FR GB IE IT NL SE
CN 1224443 A 19990728 (199948) C11B003-00
BR 9710183 A 19990810 (199953) C11B003-00
JP 2000505134 W 20000425 (200031) 25p C11B007-00
MX 9900308 A1 19990501 (200056) C11B003-00
EP 912665 B1 20010816 (200147) EN C11B003-00

R: BE DE DK ES FI FR GB IE IT NL SE
DE 69706177 E 20010920 (200163) C11B003-00
AU 739166 B 20011004 (200166) C11B003-00
ES 2160965 T3 20011116 (200201) C11B003-00
US 2001047101 A1 20011129 (200202) C07J009-00 <--
ADT WO 9801519 A1 WO 1997-EP3491 19970630; AU 9735400 A AU 1997-35400 19970630; ZA 9705992 A ZA 1997-5992 19970704; EP 912665 A1 EP 1997-931750 19970630, WO 1997-EP3491 19970630; CN 1224443 A CN 1997-196132 19970630; BR 9710183 A BR 1997-10183 19970630, WO 1997-EP3491 19970630; JP 2000505134 W WO 1997-EP3491 19970630, JP 1998-504754 19970630; MX 9900308 A1 MX 1999-308 19990105; EP 912665 B1 EP 1997-931750 19970630, WO 1997-EP3491 19970630; DE 69706177 E DE 1997-606177 19970630, EP 1997-931750 19970630, WO 1997-EP3491 19970630; AU 739166 B AU 1997-35400 19970630; ES 2160965 T3 EP 1997-931750 19970630; US 2001047101 A1 Cont of US 2000-202785 20000803, US 2001-898418 20010703
FDT AU 9735400 A Based on WO 9801519; EP 912665 A1 Based on WO 9801519; BR 9710183 A Based on WO 9801519; JP 2000505134 W Based on WO 9801519; EP 912665 B1 Based on WO 9801519; DE 69706177 E Based on EP 912665, Based on WO 9801519; AU 739166 B Previous Publ. AU 9735400, Based on WO 9801519; ES 2160965 T3 Based on EP 912665
PRAI EP 1996-201870 19960705
IC ICM **C07J009-00**; C11B000-00; C11B003-00; C11B007-00
ICS A23D009-02; A61K007-00; C11B013-00
AB WO 9801519 A UPAB: 19980406
Oryzanol (any type of steryl cinnamic **acid** derivatives) concentrate may be obtained from an oryzanol-containing **fatty** substance, which is preferably a crude oil, by (a) at least one of (1) removal of phospholipids present, and (2) removal of free **fatty acids**, preferably by stripping; (b) alkali neutralisation of the product; and (c) separation and removal of the oil phase. Also claimed is the oryzanol concentrate obtained, particularly the concentrate in fat or oil, especially rice bran oil.
USE - Oryzanol has potential pharmacological uses, in fat-based food products, and in cosmetic uses.
ADVANTAGE - No other economically feasible method of direct recovery of oryzanol from a crude oil is known. This process is highly economical. The oryzanol is obtained substantially free of any undesired components.
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B01-D02; D03-B; D03-H; **D10-A04**; **E01**; **E11-Q01**

L58 ANSWER 22 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1995-049139 [07] WPIX
DNC C1995-022431
TI Efficient removal of **cholesterol** in fat and oil-satd. hydrocarbon mixed soln. - by adding aq. cyclodextrin soln. to mixt., stirring and removing water phase.
DC **D23** E15
PA (NISP) NISSIN SHOKUHIN KAISHA LTD; (UEDA-N) UEDA SEIYU KK
CYC 1
PI JP 06330080 A 19941129 (199507)* 5p C11B003-02
JP 2726217 B2 19980311 (199815) 5p C11B003-02
ADT JP 06330080 A JP 1993-116811 19930519; JP 2726217 B2 JP 1993-116811 19930519
FDT JP 2726217 B2 Previous Publ. JP 06330080
PRAI JP 1993-116811 19930519
IC ICM C11B003-02
ICA A23C015-16
AB JP 06330080 A UPAB: 19960529
Removal of **cholesterol** in fat and oil comprises adding aq. soln. of cyclodextrin to a fat and oil-satd. hydrocarbon mixed soln. comprising

100 pts.wt. of fat and oil and 1-50 pts.wt. of a 5-10C linear satd. hydrocarbon; stirring the mixt. at a temp. equal to or higher than the m.pt. of the fat and oil; and removing water phase.

Also claimed is prepn. of low-**cholesterol** fat and oil by adding aq. soln. of cyclodextrin to a fat and oil-satd. hydrocarbon mixed soln. comprising 100 pts.wt. of fat and oil and 1-50 pts.wt. of a 5-10C linear satd. hydrocarbon; stirring the mixt. at a temp. equal to or higher than the m.pt. of the fat; and oil and collecting oil phase.

Suitable linear satd. hydrocarbons include n-pentane, n-hexane, n-heptane, n-octane, n-nonane and n-decane, of which the most pref. is n-hexane.

USE/ADVANTAGE - Methods are very efficient and feasible in a mixing and stirring time as short as 30-60 mins.

In example, methods achieved a **cholesterol** level of 0.51 mg/g and a **cholesterol** removal rate of 75.7% for the stirring time of 30 mins. and 0.40 mg/g and 81.0%, respectively, for the stirring time of 60 mins.

Dwg.0/0

FS CPI
FA AB; GI; DCN
MC CPI: D06-H02; **D10-A04**; **E01**; E06-A03; E11-Q02

L58 ANSWER 23 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1994-256615 [32] WPIX
DNC C1994-121037
TI Sepn. of tocepherol and **sterol** from, e.g. **sunflower oil distillates** - including a step involving **esterification** of the **sterols** with **fatty acids** present in the **distillates**.
DC **D23** E13
IN FIZET, C
PA (HOFF) HOFFMANN LA ROCHE & CO AG F; (HOFF) HOFFMANN LA ROCHE INC
CYC 13
PI EP 610742 A1 19940817 (199432)* DE 11p C07D311-72
R: AT BE CH DE DK ES FR GB IT LI NL
JP 07002827 A 19950106 (199511) 9p C07D311-72
US 5487817 A 19960130 (199611) 7p B01D003-34
JP 2648450 B2 19970827 (199739) 9p C07D311-72
EP 610742 B1 19990324 (199916) DE C07D311-72
R: AT BE CH DE DK ES FR GB IT LI NL
DE 59407982 G 19990429 (199923) C07D311-72
ES 2130294 T3 19990701 (199933) C07D311-72
ADT EP 610742 A1 EP 1994-101257 19940128; JP 07002827 A JP 1994-34234 19940208; US 5487817 A US 1994-185571 19940121; JP 2648450 B2 JP 1994-34234 19940208; EP 610742 B1 EP 1994-101257 19940128; DE 59407982 G DE 1994-507982 19940128, EP 1994-101257 19940128; ES 2130294 T3 EP 1994-101257 19940128
FDT JP 2648450 B2 Previous Publ. JP 07002827; DE 59407982 G Based on EP 610742; ES 2130294 T3 Based on EP 610742
PRAI CH 1993-467 19930211
IC ICM B01D003-34; C07D311-72
ICS B01D003-00; **C07J009-00**; C07J075-00
AB EP 610742 A UPAB: 19941010
Sepn. of tocopherols and **sterols** from fat **residues** from vapour sepn. for diodorisation comprises (a) **esterification** of the **sterols** in the fats with **fatty acids** which are also present; (b) **distillation** of the resulting mixt. to recover **residual fatty acids** and to recover tocopherols, leaving the **sterol esters** in the **distillation residues**; and (c) isolation of the tocepherols from the **distillate** and isolation of the **sterols**, after cleavage of the **esters**, from the **distillation residues**.

USE/ADVANTAGE - The tocopherol prods. have vitamin E activity. The process allows recovery of the tocopherols and **sterols** in separate stages, and are easier and cheaper than previous processes. Dwg.0/0

FS CPI
FA AB; DCN
MC CPI: D10-A02; **E01**; E06-A01; **E11-Q01**
ABEQ US 5487817 A UPAB: 19960315
A process for separating tocopherols and **sterols** from deodorizer sludges comprising **sterols, fatty acids,** and tocopherols, said process consisting essentially of the steps of:

(a) **esterifying** the **sterols** in the deodorizer sludge with **fatty acids** which are also present in the deodorizer sludge to form a mixture comprising **sterol fatty acid esters, fatty acids,** and tocopherols;

(b) **distilling** said mixture to obtain a first **distillation** fraction containing **fatty acid residues,** a second **distillation** fraction containing tocopherols, and a **sterol fatty acid ester residue;**

(c) isolating the tocopherols from the second **distillation** fraction; and

(d) isolating the **sterols** from the **sterol fatty acid ester residue.**

Dwg.0/0

L58 ANSWER 24 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1993-385740 [48] WPIX
DNC C1993-171533
TI Reducing amt. of **cholesterol** and hydroxy cpds. in animal fat - by treatment with succinic or glutaric anhydride.
DC D13 **D23** E15
IN CHEN, Y; HAMMOND, E G
PA (IOWA) UNIV IOWA STATE RES FOUND INC
CYC 1
PI US 5264599 A 19931123 (199348)* 5p C11B007-00
ADT US 5264599 A US 1991-790228 19911108
PRAI US 1991-790228 19911108
IC ICM C11B007-00
AB US 5264599 A UPAB: 19940120
A process (A) for treating an edible animal fat to reduce the content of components contg. free OH gps. comprises: (a) mixing the fat with succinic or glutaric anhydride in molar excess over the OH-contg. components; (b) heating the mixt. to a temp. promoting the conversion of the components to hemisuccinates or hemiglutarates; and (c) subjecting the prod. to alkaline refining to remove the converted components as water-soluble soaps.

A process (B) for treating an edible animal fat to reduce the free **cholesterol** content comprises: (a) mixing hte fat with succinic anhydride in at least a 1.2 molar excess over the free **cholesterol** ; (b) heating the mixt. to a temp. promoting the conversion of **cholesterol** to **cholesteryl** hemisuccinate (CHS); and (c) subjecting the prod. to alkaline refining to remove the CHS as a water-soluble soap.

ADVANTAGE - **Cholesterol** reductions of 40-42% have been achieved for lard, lard oil, tallow and tallow oil, and ca. 30% for butter oil.

FS CPI
FA AB; DCN
MC CPI: D03-C; **D10-A04**; **E01**; E07-A02C; E07-A02G; E11-Q02

L58 ANSWER 25 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1993-347480 [44] WPIX

DNC C1993-153704
TI Sepn. of optical isomers of methyl jasmonate and its derivs. - by HPLC using silica gel coated with amylose tris-(S)-methylbenzyl carbamate as packing material and N-hexane and 2-**propanol** mixt. as mobile phase.
DC B05 **D23** E15
PA (SUMO) SUMITOMO CHEM CO LTD
CYC 1
PI JP 05246950 A 19930924 (199344)* 5p C07C069-716
ADT JP 05246950 A JP 1992-46810 19920304
PRAI JP 1992-46810 19920304
IC ICM C07C069-716
ICS B01D015-08; C07C067-48
AB JP 05246950 A UPAB: 19931213
Sepn. comprises sepg. methyl jasmonate or methyl epijasmonate through HPLC column for sepn. of optical isomers using silica gel coated with amylose tris ((S)-methylbenzyl carbamate) as a packing substance and 9:1 mixture of n-hexane and 2-**propanol** as a mobile phase.
Also claimed is sepn. of optical isomers of methyl dihydrojasmonate and methyl cucurbinate through HPLC column using 49:1 and 19:1 mixt. of n-hexane and 2-**propanol** as a mobile phase, respectively.
USE/ADVANTAGE - Optical isomers of methyl jasmonate and its related substances, prepd. by asymmetric synthesis can be sepd. rapidly and effectively.
FS CPI
FA AB; DCN
MC CPI: B10-E04A; B10-F02; B11-B; B12-L07; **D10-A04**; D10-A05; **E01**; E10-F02A1; **E11-Q01**

L58 ANSWER 26 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1992-416319 [51] WPIX
DNC C1992-184726
TI Sepg. impurities from animal fat on pptd. silica - esp. cholest-5-en-3 beta-yl **ester**(s) and/or cholest-5-en-3e beta-ol, avoiding environmental contamination.
DC B01 **D23** E33 J01
IN GOHLISCH, F; KUESTER, M; SCHULZE, A
PA (COSW) CHEMIEWERK COSWIG; (WASC-N) WASCHMITTELWERK GENTHIN
CYC 1
PI DD 300842 A5 19920813 (199251)* 3p B01J020-10
ADT DD 300842 A5 DD 1990-337028 19900110
PRAI DD 1990-337028 19900110
IC ICM B01J020-10
AB DD 300842 A UPAB: 19931116
An adsorbent for selective sepn. of impurities from animal fats, esp. carboxylic acid cholest-5-en-3-beta-yl **esters** and/or cholest-5-en-3-beta-ol comprises finely dispersed pptd. SiO2.
The adsorbent has 80% of particle size 0.5-1.8 x 10 power-5 m. and 20% 1.8 x 10 power-5 m-2.5 x 10 power-4 m., and BET specific surface 1,5-30 sq.m/g.
ADVANTAGE - The adsorbent is a cheap and previously useless waste prod. from prepn. of AlF3 and avoids contamination of the environment.
In an example, 2.1 g. of a **distn. residue** contg. 9% of **cholesteryl ester** was dissolved in 25 cc. of CCl4 and passed at room temp. through a column of 100 g. of pptd. SiO2. Elution with CCl4 and processing gave 0.145 g. of the **cholesteryl ester** (77% yield)
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B01-D02; D10-A02; **E01**; E31-P03; J01-B

L58 ANSWER 27 OF 34 WPIX (C) 2002 THOMSON DERWENT

AN 1992-093992 [12] WPIX
DNC C1992-043786
TI Fractionation purificn. of lipid fraction - from hydrophilic organic solvent extract of animal fat and oil by centrifuging liq.-liq. partition chromatography.
DC **D23** E19
PA (SNOW) SNOW BRAND MILK PROD CO LTD
CYC 1
PI JP 04039398 A 19920210 (199212)* 5p
ADT JP 04039398 A JP 1990-146921 19900605
PRAI JP 1990-146921 19900605
IC C11B001-10; C11B003-10; C11B007-00
AB JP 04039398 A UPAB: 19931006
In a new fractionation purificn. of lipid fractions from a hydrophilic organic solvent extract of an animal fat and oil, the extract is fractionated into **fatty acid triglycerides**, **cholesterol**, phosphatidyl ethanolamine, phosphatidyl choline, sphingomyelin and/or **glyceroglycolipids** by centrifuging based liq. - liq.-partition chromatography using a mixed solvent comprising a satd. hydrocarbon(s), an alcohol(s), and water or a mixed solvent comprising a satd. hydrocarbon(s), an ether(s), an alcohol(s) and water, and the resultant fractions are purified.
Pref. the extract is first fractionated into a **fatty acid glyceride-cholesterol** fraction, a phospholipid fraction (I) based on phosphatidyl ethanolamine, a phospholipid fraction (II) based on sphingomyelin and/or **glyceroglycolipid** by the chromatography using mixed solvent comprising a satd. hydrocarbon(s) an alcohol(s) and water, and the resultant fractions are purified.
USE/ADVANTAGE - The efficient methods provide high-purity fractions, allowing recovery of ingredients in the solvent used for the removal of **cholesterol**.
0/1
FS CPI
FA AB; DCN
MC CPI: D10-A01; **D10-A04**; **E01**; E05-G09D; E10-G02B; **E11-Q01**

L58 ANSWER 28 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1992-024813 [04] WPIX
TI Sepn. of mixed **fatty acids** from deodoriser **distillate** - by melting **distillate**, adding to refluxing soln., mixing and cooling mixt. sepg. and drying crystals, dissolving and sepg. organic layer.
DC **D23** E17 E19
IN MAZA, A
PA (CORP) CPC INT INC
CYC 2
PI CA 2041110 A 19911028 (199204)*
US 5078920 A 19920107 (199205)
ADT CA 2041110 A CA 1991-2041110 19910424; US 5078920 A US 1990-515938 19900427
PRAI US 1990-515938 19900427
IC C07C051-43; C07C053-12; C07C057-03; C07D311-72; **C07J009-00**; C11B003-10
AB CA 2041110 A UPAB: 19931006
A process for separating mixed **fatty acids** from a deodoriser **distillate** comprises the sequential steps of melting the **distillate**, adding the melted **distillate** to a refluxing soln. of urea and alcohol to form a reaction mixt., mixing the reaction mixt. while cooling to allow formation of crystals, separating the crystals, drying them, dissolving them in water to form an organic layer rich in mixed **fatty acid**, and an aq. layer

contg. urea and separating the **fatty acid** layer.

The **distillate** is melted at 50-80 deg.C and the refluxing soln. of urea and alcohol is maintained at 10 deg.C below the boiling point of the alcohol to 2 deg.C above the boiling point of alcohol. The concn. of urea in the refluxing soln. of urea and alcohol is 5-75 wt% and the crystals are dried at ambient temp. An excess of 5-50% water is utilised to dissolve the crystals. The alcohol is selected from **MeOH**, **propanol**, isopropanol, **butanol**, isobutanol, t-**butanol** and sec-**butanol**. The deodoriser **distillate** is a byprod. from processing fats and **oils** from sources selected from **soybeans**, corn, **rapeseed**, cottonseed, **coconut**, **palm**, peanut, rice bran, lard and tallow. The mixed **fatty acids** are selected from butyric, caproic, caprylic, capric, lauric, myristic, mysritoleic, pentadecanoic, palmitic, palmitoleic, marganic, margaroleic stearic, oleic, linoleic, linolenic, arachidic, galloleic, eicosadienoic, behenic, erveic, docosadienoic and lignoceric. The mother liquor from the crystals are saved for processing in second stage. The second stage comprises evaporating the mother liquor to form a **residue** adding water to form organic layer rich in tocophenols and **sterols** and an aq. layer contg. urea and separating the organic layer.

USE/ADVANTAGE - The process improves the yield of mixed **fatty acids**, tocophcrols, and **sterols** separated from deodoriser **distillates** which are a byprod of the edible **oils** and fats industries. The process uses reduced amts. of organic solvents. @

0/0@

FS CPI

FA AB; DCN

MC CPI: D10-B01; **E01**; E10-C04H; E10-C04L; **E11-Q01**

ABEQ US 5078920 A UPAB: 19931006

Sepn. of mixed **fatty acids** from a deodoriser **distillate** comprises (a) melting a deodoriser **distillate** ; (b) adding melted deodoriser to a refluxing soln. of urea and alcohol to form a reaction mixt.; (c) mixing the mixt. while cooling to allow formation of crystals; (d) sepg. and drying crystals; (e) dissolving the crystals in water to form an organic layer rich in **fatty acids** and an aq. layer contg. urea; then (f) sepg. the **fatty acid** layer.

Soln. of urea and alcohol is used in a conc. of 5-75 wt.% and is pref. non-aq.. Soln. is refluxed at a pref. temp. of (-) 10 - (+) 25 deg.C based on b.pt. of alcohol. Drying is pref. carried out using ambient air. Pref. excess of water is 5-50%.

USE/ADVANTAGE - Increased yields and reduced use of organic solvents in the edible oils and fats industries. Recovered **fatty acids** are used in the making of soap, detergents and resin.

L58 ANSWER 29 OF 34 WPIX (C) 2002 THOMSON DERWENT

AN 1991-200620 [27] WPIX

DNC C1991-086839

TI Removing **sterol**(s) for lipid(s), esp. **cholesterol** from food fats - by dissolving **sterol** and lipid mixt. in high pressure fluid and contacting with adsorbent for **sterol**.

DC B01 D13 **D23** E15

IN CATCHPOLE, O J; HAMILTON, B H; MCLACHLAN, C N S

PA (MCLA-I) MCLACHLAN C N S

CYC 3

PI US 5024846 A 19910618 (199127)*

NZ 221586 A 19930225 (199312) A23C015-00

CA 1320948 C 19930803 (199337) C07J009-00 <--

ADT US 5024846 A US 1990-561477 19900802; NZ 221586 A NZ 1987-221586 19870826; CA 1320948 C CA 1988-564495 19880419

PRAI NZ 1987-221586 19870826

IC A23C015-16; A23D007-02; C11B003-06; C11B003-10; C11B007-00
ICM **C07J009-00**
ICS A23C015-12; A23C015-16; A23D007-02; C11B003-06; C11B003-10; C11B007-00
AB US 5024846 A UPAB: 19930928
Method of sepg. **sterols** from lipids includes: (a) dissolving a **sterol**/lipid mixt. in a high pressure physiologically acceptable fluid from a high pressure liq., a high pressure subcritical gas or a high pressure supercritical gas, to form a high pressure fluid mixt.; (b) contacting this mixt. with an adsorbent material comprising oxygen-contg. salts of the basic metals to adsorb the **sterols** selectively; and (c) removing the **sterol**-free lipids from the high pressure fluid.
USE/ADVANTAGE - Esp. useful for removing **cholesterol** from animal/ **plant oils**, meat, cheese, milk fats, egg powder and esp. butter. Extraction conditions are such that organoleptic properties of the food prod. are not effected. The adsorbents are relatively inexpensive and can be regenerated, and the high pressure fluid may be recycled. Process could also be applied to concentrate e.g. hormones, steroids, vitamin D.
0/2
FS CPI
FA AB; DCN
MC CPI: B01-D02; B03-G; B04-B02D; D03-B; D03-C; D03-H01T; **E01**; **E11-Q01**; E31-K05B; E31-K05C; E34; E35

L58 ANSWER 30 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1990-115977 [15] WPIX
DNC C1990-050963
TI Removal of **sterol**(s) from lipid(s) - by soln. in high pressure fluid, and treatment with an inorganic adsorbent.
DC B01 D13 **D23** E15
IN CATCHPOLE, O J; HAMILTON, B H; MCLACHLAN, C N S
PA (MCLA-I) MCLACHLAN C N S; (PIPE-I) PIPER J W
CYC 30
PI WO 9002788 A 19900322 (199015)*
RW: AT BE CH DE FR GB IT LU NL OA SE
W: AU BB BG BR DK FI HU JP KP KR LK MC MG MW NO RO SD SU
AU 8823865 A 19900402 (199025)
FI 9101168 A 19910308 (199123)
BR 8807920 A 19910521 (199125)#
NO 9100926 A 19910308 (199126)
EP 434676 A 19910703 (199127)
R: AT BE CH DE FR GB IT LI LU NL SE
DK 9100418 A 19910429 (199130)
JP 05504975 W 19930729 (199335) 9p C11B003-10
AU 9346297 A 19931202 (199404)# C11B003-10
HU 65567 T 19940728 (199431) C11B003-10
AU 657233 B 19950302 (199516)# C11B003-10
ADT EP 434676 A EP 1988-907756 19880909; JP 05504975 W JP 1988-507368 19880909, WO 1988-GB739 19880909; AU 9346297 A AU 1993-46297 19930910, Div ex AU 1988-23865 ; HU 65567 T HU 1988-5530 19880909, WO 1988-GB739 19880909; AU 657233 B AU 1993-46297 19930910, Div ex AU 1988-23865
FDT JP 05504975 W Based on WO 9002788; HU 65567 T Based on WO 9002788; AU 657233 B Previous Publ. AU 9346297
PRAI WO 1988-GB739 19880909
REP US 4049688; US 4692280; US 4734226; WO 8802989
IC C11B003-10
ICM C11B003-10
ICS C11B007-00
AB WO 9002788 A UPAB: 19930928
Sterols (I) are sepd. from lipids (II) as follows: (a) the (I)/(II) mixt. is dissolved in a high pressure, non-toxic fluid (III) (as a

high pressure liq. or as a high pressure sub- or supercritical gas); (b) the high pressure mixt. is contacted with an adsorbent (IV) so that (I) are selectively adsorbed; and (c) the **sterol**-free lipids are removed from the high pressure fluid. (IV) is made up from or includes O-contg. salts of basic metals such as (hydr) oxides, carbonates, or sulphates, etc.

Uses sub- or supercritical CO2 at 30-60 deg.C/50-400 Bar. Pref. (IV) is Ca(OH)2, CaO, CaCO3, MgCO3, and Mg(OH)2.

ADVANTAGE - The process removes **sterols** from lipids without modify ng the flavour, physical properties, or keeping properties of the latter. The process is useful for the removal of **cholesterol** from butter. @

0/2

FS CPI
FA AB; DCN
MC CPI: B01-D02; B04-B01B; B04-B02D; B11-B; D03-B; **E01**; **E11-Q01**
ABEQ JP 05504975 W UPAB: 19931119

Sterols (I) are sepd. from lipids (II) as follows: (a) the (I)/(II) mixt. is dissolved in a high pressure, non-toxic fluid (III) (as a high pressure liq. or as a high pressure sub- or supercritical gas); (b) the high pressure mixt. is contacted with an adsorbent (IV) so that (I) are selectively adsorbed; and (c) the **sterol**-free lipids are a removed from the high pressure fluid. (IV) is made up from or includes O-contg. salts of basic metals uch as (hydr) oxides, carbonates, or sulphates, etc..

Uses sub- or supercritical CO2 at 30-60 deg. C/50-400 Bar. Pref. ((V) is Ca(OH)2, CaO, CaCO3, MgCO3, and Mg(OH)2.

ADVANTAGE - The process removes **sterols** from lipids without modifying the flavour, physical properties, or keeping properties of the latter. The process is useful for the removal of **cholesteroal** from butter.

L58 ANSWER 31 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1989-364567 [50] WPIX
DNC C1989-161682
TI Crude **fatty acid** fractionation - with **sterol** enrichment in bottoms prod..
DC **D23** E15
IN GASSER, G; GUTWASSER, H; VOSS, B
PA (OLMA-N) VEB OL & MARGARINE
CYC 1
PI DD 269859 A 19890712 (198950)* 9p
ADT DD 269859 A DD 1987-311913 19871231
PRAI DD 1987-311913 19871231
IC **C07J009-00**; C11C001-10
AB DD 269859 A UPAB: 19930923

Fractionation of crude **fatty acids** is effected by: (a) preheating the feed in a heat exchanger with steam at 0.2-0.5 (0.3) MPa gauge, and heating it to boiling temp. at 2.5-20 (10) KPa in a heat exchanger with a heat-transfer fluid ; (b) degassing and drying the feed at 2.5-3.5 (3) KPa in a 1st vertical tube-and-shell (VTS) heat exchanger, and heating it at 400-1500 (500) Pa in a 2nd VTS heat exchanger with steam at 3.2-4.4 (3.8) MPa gauge; (c) evaporating the feed in a 1st falling-film (FF) evaporator, r evaporting the liq. phase in a 2nd FF evaporator at 300-600 (400) Pa, and stripping the bottom stream in a natural-circulation (NC) evaporator to obtain a **sterol**-enriched bottoms fraction, which is cooled in a heat exchanger from the system; (d) passing the vapour streams (DI) from the 2nd VTS heat-exchanger, the two FF evaporators and the NC evaporator through the outer zone (I) of a double-flow tube-and-shell heat exchanger in countercurrent to liq. streams (F1-F3), and recycling the **sterol**-rich condensate (K1) to the 2nd FF evaporator; (e) condensing the **sterol**-free vapour

from the outer zone in two condensers, heating the condensate to boiling in a preheater (14) with steam at 3.2-4.4 (3.8) MPa gauge, and passing it (F2) through a 1st section of the inner zone , the pressure being 200-500 (300) Pa in the outer zone and 50-150 (133) Pa in the inner zone; (f) passing the condensate (K2) from the outer zone to a cooler and discharging obtd. **fatty acid** prod. fraction from the system; (g) condensing the vapour from the 1st section of the inner zone in two condensers, heating condensate to boiling temp. at 100-500 (150) Pa in a preheater (19) with steam at 3.2-4.4 (3.8) MPa gauge, and passing it (F1) to the 2nd section of the inner zone; and collecting the final prod..

USE - **Distn.** of crude **fatty acids**.

0/1

FS CPI
FA AB; DCN
MC CPI: D10-B01; E10-C04J; E10-C04K; **E11-Q01**

L58 ANSWER 32 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1989-243247 [34] WPIX
DNC C1989-108286
TI Removal of steroid cpds. from fats and oils - by extn. with solvent system pref. contg. **methanol**.
DC B01 D13 **D23** E15
IN HOBMAN, P G; KEEN, A R; WARD, D D
PA (NZDA-N) NEW ZEALAND DAIRY RES INST; (NZDA-N) NZ DAIRY RES INST
CYC 16
PI EP 329347 A 19890823 (198934)* EN 11p
R: AT BE CH DE ES FR GB IT LI LU NL SE
AU 8929888 A 19890817 (198941)
ZA 8901082 A 19891025 (198948)
JP 02008297 A 19900111 (199008)
CA 1332056 C 19940920 (199438) C07J009-00 <--
ADT EP 329347 A EP 1989-301304 19890210; ZA 8901082 A ZA 1989-1082 19890210; JP 02008297 A JP 1989-31239 19890213; CA 1332056 C CA 1989-590794 19890210
PRAI NZ 1988-223508 19880212
REP 1.Jnl.Ref; A3...9036; FR 2097322; No-SR.Pub; SU 950393
IC A23C015-00; A23C019-00; A23D005-00; B01D000-00; C11B003-00
ICM **C07J009-00**
ICS A23C015-00; A23C019-14; A23D005-00; B01D000-00; C11B003-00
AB EP 329347 A UPAB: 19930923
The amt. of steroidal cpds. (I) (**sterols** and other cpds.) present in edible fats and/or oil is reduced by extrn. with a solvent system; (I) may then be recovered from the sepd. solvent(s).

Pref. sepn. is for the removal of **cholesterol** from cream and/or other aq. emulsions or colloidal suspensions of fats and/or oils. Extn. is pref. at a temp. sufficient to liquify the fat or oil, with ratio solvent : fat or oil = 5:1. Pref. solvent systems are **MeOH** and aq. **MeOH**. Solvent may be recovered by known methods, including passage over a C adsorbent, passage through a reverse phase adsorption column, reverse osmosis, or by solvent extn. (e.g., CHCl3/H2O).

USE/ADVANTAGE - The process is effective in the selective removal of **sterols** and other steroid cpds. from edible fats and/or oils. The method is esp. useful for the prepn. of a **cholesterol**-reduced milk fat (possessing most of the natural colour and flavour) which may be used in the processed food industry.

0/0

FS CPI
FA AB; DCN
MC CPI: B01-D02; B04-B01B; B04-B04K; B11-B; D03-B; D03-H01C; D10-A01; **E01; E11-Q01**

L58 ANSWER 33 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1987-318213 [45] WPIX

DNC C1987-135679
TI Extracting and sepg. steroid from fat material - by saponifying with alkali and contacting with supercritical gas.
DC B01 **D23** E17
PA (HITA) HITACHI LTD
CYC 1
PI JP 62226997 A 19871005 (198745)* 7p
JP 06080073 B2 19941012 (199439) 8p C07J075-00
ADT JP 62226997 A JP 1986-67346 19860327; JP 06080073 B2 JP 1986-67346 19860327
FDT JP 06080073 B2 Based on JP 62226997
PRAI JP 1986-67346 19860327
IC B01D011-00; D07J075-00
ICM C07J075-00
ICS B01D011-00; D07J075-00
AB JP 62226997 A UPAB: 19930922
Method comprises adding alkali to material until saponified, and contacting the material with supercritical gas.
USE - For making valuable unsatd. **fatty acids.**
0/0
FS CPI
FA AB; DCN
MC CPI: B04-B01B; B04-B02D; D10-A02; **E01**; **E11-Q01**

L58 ANSWER 34 OF 34 WPIX (C) 2002 THOMSON DERWENT
AN 1987-218365 [31] WPIX
DNC C1987-091998
TI Separation and purification of **cholesterol** - by **distilling** fish oil and crystallising, used for fish feed additive or pharmaceutical or cosmetic ingredient.
DC B01 C03 D13 D21 **D23** E15
PA (NIUS) NIPPON SUISAN KAISHA LTD
CYC 1
PI JP 62145099 A 19870629 (198731)* 3p
ADT JP 62145099 A JP 1985-286721 19851219
PRAI JP 1985-286721 19851219
IC **C07J009-00**; C07J075-00
AB JP 62145099 A UPAB: 19930922
Cholesterol is separated and purified by subjecting fish oil to molecular **distn.** and crystallising resultant volatile component with solvent. As solvent, organic solvent, which may be hexane, **methanol**, acetone or ethyl acetate, is used.
USE/ADVANTAGE - **Cholesterol** sepd. from fish oil is used as additive to feed for fish-culture, or ingredient for cosmetics, pharmaceuticals or for liq. crystals.
In an example, degumm-treated coasted sardine crude oil (acid value 3.8, nonsaponified substance content 2.0%, **cholesterol** content 1.8%) (15 kg) was subjected to molecular **distn.** using centrifugal molecular **distn.** appts. MS 380 type heating at temp. of 220 det.C sample oil rate of 20 L/H, Petri dish rotation of 2,000 rpm, and vacuum of 5 x 10 power (-3) mmHg. As the result, diacidified sardine oil (14.5 kg) as **residual** fraction and oily substance, semisolid at normal temp. (0.45 kg, **cholesterol** content 35.0%) were obtd.
0/0
FS CPI
FA AB
MC CPI: B01-D02; B12-L02; B12-L09; C01-D02; C12-L02; C12-L09; D03-G; D08-B; D10-A01; **E01**; **E11-Q01**

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(FILE 'HOME' ENTERED AT 07:31:40 ON 13 AUG 2002)

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                SET COST OFF

     FILE 'WPIX' ENTERED AT 07:31:49 ON 13 AUG 2002
                E BONAKDAR M/AU
L1            7 S E3
                E WOLLMAN G/AU
L2            2 S E3
L3           37 S E22
                E GUTSCHE B/AU
L4           63 S E3
                E GUETSCHE B/AU
                E GEUTSCHE B/AU
                E SCHWARZER J/AU
L5           21 S E3
                E COGNIS/PA
L6          860 S E3-E18
L7          934 S L1-L6
L8        14154 S ?STEROL?
L9           35 S L7 AND L8
                E SCHWAZER J/AU
L10           1 S E3
L11          35 S L7,L10 AND L8
L12          35 S L9,L11
L13           1 S L1 AND L2-L5,L10
L14          15 S L2,L3 AND L4,L5,L10
L15           5 S L4 AND L5,L10
L16           1 S L13 AND L14,L15
L17           2 S L14 AND L15
L18           2 S L16,L17
L19           2 S L18 AND L1-L18
L20         202 S (Q271(S)Q431)/M0,M1,M2,M3,M4,M5,M6
L21       21142 S "E11-Q01"/MC
L22         474 S D10-A04/MC
L23        1819 S "E01"/MC
L24        1872 S C07J009/IC,ICM,ICS
L25           4 S L23,L24 AND L20
L26          51 S L23,L24 AND L21
L27          15 S L23,L24 AND L22
L28          57 S L25-L27
L29         797 S D23/DC AND L20,L21,L22
L30          30 S L29 AND L28
L31          30 S L19,L30
L32          27 S L28 NOT L31
                SEL DN AN 2 5 6 7 8 9 10 24
L33           8 S L32 AND E1-E16
L34          38 S L31,L33
L35       14410 S (TALL OR SOYBEAN OR SOY# BEAN OR SOJABEAN OR SOJA BEAN OR SOY
L36       13532 S VEGETABLE(L)OIL
L37       11843 S PLANT(L)OIL
L38          20 S L34 AND L35,L36
L39           4 S L34,L38 AND (TRANSESTER? OR TRANS ESTER?)
L40          27 S L34,L38 AND ?ESTER?
L41          19 S L34,L38 AND FATTY(L)ACID
L42          10 S L34,L38 AND DISTILL?(L)RESIDU?
L43           8 S L34,L38 AND LOWER(L)ALC?
L44          16 S L34,L38 AND (METHANOL OR ETHANOL OR PROPANOL OR BUTANOL OR ME
L45           7 S L34,L38 AND ?GLYCER?
L46           5 S L34,L38 AND (0270 OR 0245 OR 0302 OR 0304 OR 0436 OR 0373)/DR
L47           4 S L34,L38 AND (R00270 OR R00245 OR R00302 OR R00304 OR R00436 O
L48          38 S L34,L38,L39-L47
L49           4 S L19,L39 AND L48
L50          18 S L34,L38 AND DISTILL?
L51           3 S L49 AND L50
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L52           4 S L49,L51
L53          34 S L34,L38,L48-L51 NOT L52
                SEL DN AN 17 21 22 24 25
L54          29 S L53 NOT E17-E26
L55          34 S L53,L54 AND L1-L54
L56          16 S L55 AND L35
L57          17 S L55 AND (TALL OR SOYBEAN OR SOY# BEAN OR SOJABEAN OR SOJA BEA
L58          34 S L55-L57

     FILE 'WPIX' ENTERED AT 08:10:26 ON 13 AUG 2002
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